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# A COURSE IN FOOD ANALYSIS

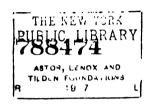
# ANDREW L. WINTON, Ph.D.

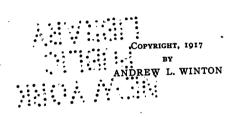
AUTHOR OF THE MICROSCOPY OF VEGETABLE FOODS; REVISER OF LEACH'S FOOD INSPECTION AND ANALYSIS; TRANSLATOR OF HANAUSEK'S MICROSCOPY OF TECHNICAL PRODUCTS

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# **PREFACE**

THE purpose of this book is first to start the chemical student on the right road to the intelligent use of more extensive works and thereby become a professional food analyst, and second, to meet the needs of the general student who takes up food analysis partly for mental and manual discipline and partly because of its bearing on subjects such as agriculture, food manufacture, nutrition, and household economics. Although the detailed instructions may seem more adapted to the wants of the student of the second class, whose training may have been limited to class room and laboratory work in general chemistry, it is believed that no one will find them too explicit.

The fact that a course in qualitative analysis requires a full semester of laboratory work and an abridged course in quantitative analysis another semester deters many who would otherwise avail themselves of the excellent systematic training these subjects afford. Such students may find that an introductory course in food analysis, requiring but forty laboratory periods such as this book contemplates, furnishes not only the requisite discipline, but also a general insight into the composition and microscopic structure of products needed in everyday life.

While inorganic methods have a certain degree of sameness, being largely based on precipitation or titration, the methods of food analysis include extraction, polarimetric, colorimetric, centrifugal, and distillation processes, thus furnishing training in versatility. Although the methods selected are but a few of those in the literature, they are the ones most generally used and least liable to become obsolete through change in trade practices or official rulings. After they have been thoroughly



mastered the analyst should be able to undertake at once the bulk of the work of most food laboratories. In order that he may have a clear conception of the whole subject and be able to use intelligently the literature, the principles of other important methods are briefly considered.

As some of the apparatus is not ordinarily found in the analytical laboratory care has been taken in describing it so that it can be accurately specified in ordering from the dealer. Lists of apparatus, reagents, and materials for analysis, required for the course, are given in the appendix.

While the chapters are arranged in their logical sequence, thus seeking gradually to develop the subject and bring out clearly general principles, a rigid adherence to this order by all the members of a large class would necessitate the duplication of expensive apparatus. To meet this difficulty the matter has been so arranged that it can be divided into five sections, each of which can be assigned to a group of six students, and thus one saccharimeter, one refractometer, one Westphal balance, one tintometer, one calorimeter, one polarizing microscope, six ordinary microscopes, and certain pieces of multiple apparatus be made to do duty for a class of thirty students.

Although the laboratory work may seem at first sight more than can be carried out in the time allowed, the author knows from experience that with reasonable diligence on the part of the student it can be accomplished in a satisfactory manner provided he is not called upon to prepare reagents or standardize solutions.

As an example in ethics too often neglected, if for no other reason, care has been taken in describing methods to give the names of authors and original references, although unnecessary foot notes have been avoided. Analyses of typical foods have been drawn from the compilations of Atwater and Bryant, Doane and Lawson, Farrington and Woll, Jenkins and Winton, and Koenig, also from the bulletins of Frear, Given, and Broomell, Merrill and Mansfield, and others. The constants of fats and oils are largely those given by Lewkowitsch.

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Grateful acknowledgment is also due the author's friends Dr. C. A. Browne, Prof. E. M. Chamot, Prof. T. F. Hanausek, Prof. Josef Moeller, and others for the use of cuts. Free use has been made of matter in Leach's Food Inspection and Analysis, both that inserted by Mr. Leach during his lifetime and by the author in his revisions. The efforts of friends are thus again joined in the same cause.

Wilton, Conn., March, 1917.

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# A COURSE IN FOOD ANALYSIS

### CHAPTER I

#### INTRODUCTION

Foods are classified as animal, vegetable, and mineral, and are divided into subgroups according to their source or method of manufacture, factors which are intimately correlated with their chemical composition.

Animal Foods. The Natural Animal Foods include milk. eggs, meat, and fish. All of these contain (1) Water, or moisture, (2) Crude Fat (or more correctly Ether Extract), (3) Protein (nitrogenous substances such as casein of milk, albumin of eggs, and myosin of meat), (4) Ash, or mineral matter (chiefly sodium, potassium, calcium, magnesium, and iron, combined as phosphates, sulphates, chlorides, and carbonates or in organic combinations), and (5) Carbohydrates (lactose of milk, glycogen of meat, etc.). Except in a few foods such as milk and liver, the amount of carbohydrates is so small that for ordinary purposes it is negligible. Minor constituents, such as Citric Acid of milk, Lecithin (phosphorized fat) of eggs, Zoosterols (cholesterol, etc.) of fats, Creatine, Creatinine, and Xanthine Bases of meat, although of great interest to the physiological chemist, are of comparatively small importance to the food analyst engaged in nutrition or inspection work.

Manufactured Animal Foods. Of the dairy products, cream is milk with extra fat, cheese is milk with most of the lactose (milk sugar), the albumin (soluble protein), and part of the water and mineral matter eliminated and salt added, and butter

is the fat of milk with some water, a trace of casein and mineral latter, and added salt. Eggs are usually bought for the consumer in the shell, although the shell contents, dried or frozen, and egg albumin are prepared in considerable amount from the tracked and otherwise damaged eggs which accumulate at shipping centers. Meat products include sausage and other minced foods, lard and edible tallow, which for convenience are usually treated in connection with vegetable fats and oils, gelatin, a substance related to the proteins obtained from hoofs, hides, etc., and meat extracts consisting in large part of flavoring and stimulating substances. Fish products are relatively unimportant.

Salt and wood smoke are time-honored preservatives for meat and fish. Chemical preservatives (formaldehyde, borax, boracic acid, sulphites, and sodium benzoate) of late years, have come into use, not only in meat and fish, but also in milk and dairy products. Artificial colors and in the case of canned goods metallic contamination are also met with.

Vegetable Foods. The Natural Vegetable Foods are classified as cereals, legiminous seeds, oil seeds, nuts, vegetables, fruits, spices, and alkaloidal products (tea, coffee, and cocoa). The constituents of the are divided into six groups: (1) Water, (2) Crude Fat (et) -extract), (3) Crude Fiber (cellulose, lignin, etc.), (4) Crude otein, (5) Ash, and (6) Nitrogen-free Extract (starch, sugars, gums, organic acids, etc.). All the groups but the third are common to both animal and vegetable daids although the nitrogen-free extract, which forms the bulk of the cereals, leguminous seeds, and many other vegetable foods, is a minor constituent of most animal foods excepting milk. The third group, crude fiber, is characteristic of vegetable organisms. It forms the frame work of vegetable cells consisting in the young or active tissues of Cellulose and in hardened or voody parts of cellulose and "infiltrated" substances such as Lignin (woody substance), Suberin (cork substance), Cutin ( uticle substance), etc.

More or less satisfactory methods are available for the detern ination of certain of the individual compounds present in each of the six groups as, for example, the different acids and bases of the ash, the individual proteins and the Amido Compounds of the crude protein, the volatile and non-volatile acids, the non-saponifiable matter, including Phytosterols (sitosterol, etc.) of the ether extract, and the Starch, Sugars, Pentosans, Dextr. 1, and Organic Acids of the nitrogen-free extract. Some of the most important of these methods are described in the chapters which follow.

Spices contain all the six groups of substances enumerated but are valuable only for certain *Essential Oils* or other flavoring constituents. Although soluble in ether essential oils are not grouped with the fixed (non-volatile) or fatty oils. Being volatile they pass off with the water on heating, although more slowly.

Alkaloidal foods, unlike spices, are valuable partly for their flavoring constituents but chiefly for their stimulating principles, *Caffeine* and *Theobromine*, which like essential oils can be quantitatively determined.

Manufactured Vegetable Foods are grouped as (1) cereal products (flour, meal, and other mill products), (2) leguminous products (pea meal and peanut butter), (3) all cakes (corton seed, linseed, and other cakes used, chi dy as cattle foods), (4) vegetable products (pickles, tsup, and canned vegetables), (5) fruit products (jams, je. 28, fruit juices, dried and canned fruits), (6) oils and fats, (7) saccharine products (sugar, syrup, honey, and confectionery), (8) alcoholic liquors (fruit juices, cereal extracts, or other saccharine liquids which have been fermented and in certain cases distilled), (9) vinc as (alcoholic liquors subjected to acetous fermentation, whereby alcohol oxidizes to acetic acid), and (10) flavoring extracts (alcoholic solutions of essential oils and other substances).

In the cereal, leguminous, oil seed, vegetable, and certain fruit products determinations of the six groups of constituents already dwelt on are of chief importance; in oils and fats the so-called chemical and physical constants, including Iodine Number, Saponification Number, Volatile Fatty Acias, Specific Gravity, Refraction, etc., are determined for purposes

of identification; in saccharine products determinations of sugars are made by polariscopic and chemical methods; in alcoholic liquors and vinegars Alcohol and Acetic Acid respectively are most often estimated; and in flavoring extracts the amounts of essential oil, Vanillin, or other aromatic constituents are of importance.

In addition to the chief constituents certain others of minor importance are often determined solely as a means of detecting foreign admixture. Added colors, preservatives, and metallic impurities must often be looked for.

Chemical analyses of ground or pulped vegetable substances serve only to a limited extent in determining their identity or purity. Microscopic examination supplies this deficiency. Each seed, fruit, root, bark, leaf, and flower consists of more or less characteristic tissues and cell contents which can be found under the microscope no matter how finely the material may be pulverized.

Mineral Foods. Although there are a number of mineral substances essential for animal life most of these are present in sufficient amount in animal and vegetable foods. Salt is the one exception.

Baking powders are semi-mineral foods. They are not, however, used for any purpose but to generate carbon dioxide gas, which passes off in baking, leaving behind the fixed products of the reaction.

Calculation of Calories. The function of foods is partly to repair the tissues, for which purpose proteins and mineral salts are of chief importance, and partly to furnish fuel for muscular energy and animal heat. The fuel value is expressed in calories, the unit being the heat required to raise one kilogram of water 1° C.

The calories of a given food may be determined by actual combustion in a delicate piece of apparatus known as the *Bomb Calorimeter*, or else by calculation from the analysis. Rubner uses in his calculations for one gram of each of the three classes of nutrients, carbohydrates, proteins, and fats, the factors 4.1, 4.1, and 9.3, respectively. Calculated to a pound (453.6)

453°

grams), the fuel value of the carbohydrates and proteins is 1860 calories each and of the fats is 4218 calories.

Further details with regard to calories and protein requirements will be found in the works on human nutrition and cattle feeding mentioned on p. 6).

Province and Limitations of Food Analysis. Notwithstanding the endless number of chemical compounds contained in foods, the accurate determination of only a limited number is possible with our present knowledge. These limitations of food analysis do not seriously detract from its value in the study of nutrition, in the identification and commercial valuation of foods, and in the detection of adulteration. A few determinations such as crude fat, crude fiber, crude protein, ash, nitrogen-free extract, sugar, alcohol, acids, chemical and physical constants, flavoring principles, and alkaloidal substances are sufficient for most practical purposes, while the estimation of a limited number of minor constituents serves for finer distinctions.

For example the determination of the fat (total glycerides) of milk enables the student of dietetics or nutrition to form his estimate of the food value due to fats, the dairyman to estimate the amount of butter obtainable from the milk, and the food inspector to decide whether or not the milk has been skimmed. Again the chemical and physical constants of a fat or oil enable the commercial or inspection chemist to establish its identity or purity without a detailed analysis giving the percentages of the different glycerides, were such an analysis possible.

Literature of Food Analysis. Food analysis has come into special prominence in the past generation. During this time scientific journals have been established in the leading countries, numerous articles have been published in these and other journals, and standard works have been written which in some cases have gone through several revisions.

Foods in General. The following works in the English language deal especially with the composition and analysis of all classes of food: Blyth, "Foods, Their Composition and Analysis"; Leach, "Food Inspection and Analysis"; Leff-

mann and Beam, "Select Methods of Food Analysis"; Woodman, "Food Analysis, Typical Methods and Interpretation of Results." Allen's "Commercial Organic Analysis" (Edited by Leffmann and Davis) devotes sections to the analysis of different classes of foods such as, for example, Dairy Products, (Leffmann, Revis and Bolton, Van Slyke); Meat and Meat Products (Richardson); Fats and Oils (Mitchell, Archbutt, Revis and Bolton); Sugars, Starch and its Isomers (Armstrong); and Alcoholic Liquors (Baker, Jones, Schlichting, Leffmann). The Association of Official Agricultural Chemists publishes from time to time the methods of analysis adopted by that body.

Dairy Products. Farrington and Woll, "Testing Milk and Its Products"; Richmond, "Dairy Chemistry"; Van Slyke, "Modern Methods of Testing Milk and Milk Products."

Oils and Fats. Gill, "A Short Handbook of Oil Analysis"; Lewkowitsch, "Chemical Technology and Analysis of Oils, Fats and Waxes."

Saccharine Products. Browne, "A Handbook of Sugar Analysis"; Long's translation of Landolt, "Optical Rotation of Organic Substances"; Rolfe, "The Polariscope in the Chemical Laboratory"; Weichmann, "Sugar Analysis."

Works on Food Technology. Food analysis is but a hand-maiden of more comprehensive subjects, such as food technology, nutrition, and food inspection. Of special value to the student interested in the agricultural, manufacturing, commercial, and sociological aspects of foods are the following: Bailey, "The Source, Chemistry, and Use of Food Products"; Tibbles, "Foods, Origin, Manufacture, and Composition."

Works on Nutrition. Among the works dealing especially with human nutrition are the following: Jordan, "Principles of Human Nutrition"; Lusk, "The Science of Nutrition"; Sherman, "Chemistry of Food and Nutrition"; Snyder, "Human Foods and Their Nutritive Value." The nutrition of farm animals is treated in Armsby's "Principles of Animal Nutrition."

Analyses of Foods will be found in Atwater and Bryant's "Chemical Composition of American Food Materials" and

Jenkins and Winton's "Compilation of Analyses of American Feeding Stuffs," both published by the Office of Experiment Stations of the U. S. Department of Agriculture.

Outline of the Laboratory Work. The pages which follow give general information as to the composition of the principal foods, explicit instructions for determining the most important constituents which can be carried out in 40 laboratory periods of 4 hours each, and brief statements as to other methods which, because of their unimportance or complicated nature, need not be undertaken by the novice.

The subjects considered are arranged in 8 chapters, the practical laboratory work in each chapter requiring from 2 to 8 laboratory periods.

Chapter II (4 periods) describes the determination of solids and fat in milk by different methods and tests for preservatives, the methods being those commonly followed in valuation and inspection. Methods for water, fat, salt, and curd in butter are also taken up. Cheese analysis is discussed.

Chapter III (2 periods) considers briefly the analysis of meat products and describes a method of determining the preservative sulphurous acid.

In Chapter IV (8 periods), the determination of water, fat, crude fiber, ash, and nitrogen-free extract in ground vegetable substances, also of starch in flour are treated at some length and the detection of the ingredients of baking powder is considered.

The microscopic identification of ground vegetable substances is taken up in Chapter V (8 periods).

Chapter VI (4 periods) describes the polariscopic method of determining sucrose and other sugars in saccharine products and takes up the detection of adulterants in maple products and of colors in confectionery.

The practical work in Chapter VII (4 periods) includes the determination of the principal constants of fats and oils, namely specific gravity, refractive index, iodine number, saponification number, and volatile fatty acids. It also describes the qualitative tests for cotton seed and sesame oils.

Chapter VIII (2 periods) traces analytically the transition of

sugar in fruit juices to alcohol and finally into vinegar and considers the general analysis of fruit juices, alcoholic liquors, and vinegar.

Chapter IX (5 periods) is devoted to the analysis of flavoring extracts, the practical work including the determination of vanillin, coumarin, normal lead number, and color of pure and imitation vanilla extract, also lemon (essential) oil and citral (the oxygenated flavoring constituent) of lemon extract.

Finally, Chapter X (3 periods) takes up the determination of caffeine in coffee, which is also the active principle of tea and, together with theobromine, of cocoa products. It also discusses other constituents of alkaloidal foods and their determination.

Suggestions for Division of Class. As has been noted in the preface it may be desirable to divide the subject matter and the class into groups, thus avoiding duplication of expensive apparatus. No little thought has been devoted to this feature of the book. In the author's experience, multiple pieces of apparatus, such as Kjeldahl digestion and distilling stands, water determination apparatus, and centrifugal machines are most convenient when arranged for twelve determinations, that is, for duplicate determinations of six students. That number of students can also to best advantage use on the same day apparatus such as the polariscope, refractometer, Westphal balance, etc. Accordingly it has seemed wise to provide for the division of the class into five groups of six students each and for the division of the laboratory work also into five groups of methods, taking care that each group requires the same number of laboratory periods, namely, 8. This plan is readily carried out by assigning to the first group of students, Chapters II and VII, to the second group, Chapters III, VI, and VIII, to the third, Chapter IV, to the fourth, Chapter V, and to the fifth, Chapters IX and X. At the end of the eighth laboratory period each group of students is assigned a new group of methods and so on.

If more than one student is assigned to a balance there will be less interference if each is from a different group.

Use of Balance, Burettes, etc. No attempt has been made to go into the details of construction or the method of using the pieces of apparatus found in every analytical laboratory. If the student is not familiar with them it is assumed that the instructor will arrange for extra periods devoted to such details which will naturally extend the time somewhat beyond that allowed for the course.

It is also assumed that reagents and standard solutions will be prepared, and the latter also standardized for the class. Those who have taken a course in quantitative analysis will have had this experience.

Sections Devoted to Laboratory Work. The subject matter of the book is of two kinds: (1) detailed instructions for laboratory work and (2) general information bearing on the nature, composition, and analysis of foods, including brief statements of principles involved in methods other than those carried out by the student. One without the other would be of little value. The chemist who merely learns the mechanical details of analytical methods can hardly hope to rise above the grade of a routine subordinate; on the other hand the human encyclopædia of chemical knowledge with untrained hands is of even less credit to the profession.

Notwithstanding the equal importance of the two kinds of subject matter it has seemed desirable to indicate exactly what sections deal with details of laboratory practice to guide both the instructor and the student in arranging their time to best advantage. For this purpose a five-pointed star (\*) at the left of the sideheading is used. Matter other than that starred can be made the subject for recitations.

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# CHAPTER II

# DAIRY PRODUCTS

# MILK

Composition of Milk. Milk, as it is the sole means of sustenance of the young animal, must be a perfect food, that is, it must contain all the food elements essential for life and in the proper proportion. That different animals are furnished by nature with different proportions of the different food elements appears from the following table:

AVERAGE COMPOSITION OF THE MILK OF DIFFERENT ANIMALS (KÖNIG)

	Woman's Milk.	Cow's Milk.	Goat's Milk.
Fat	3.78	3.64	4.78
Casein	1.03	3.02	3.20
Albumin	1.26	0.53	1.09
Ash	0.31	0.71	0.76
Lactose	6.21	4.88	4.46
Total solids	12.59	12.78	14.29
<b>W</b> ater	87.41	87.22	85.71
	100.00	100.00	100.00

The variation in the milk of different breeds of cows is shown in the following table:

Average Composition of the Milk of Different Breeds of Cows (Collier)

	Holstein- Friesian.	Ayrshire.	Devon.	American Holder- ness.	Jersey.	Guernsey.
Fat	3.46	3 · 57	4.15	3 · 55	5.61	5.12
Casein and albumin	3.39	3 · 43	3.76	3.39	3.91	3.61
Ash	0.73	0.69	0.76	0.70	0.74	0.75
Lactose	4.84	5 · 33	5.07	5.01	5.15	5.11
Total solids	12.42	13.02	13.74	12.65	15.41	14.59
Water	87.58	86.98	86.26	87.35	84.59	85.41
	100.00	100.00	100.00	100,00	100.00	100.00

Colostrum. The foregoing tables of composition do not take into account the abnormal milk, known as colostrum, produced for two or three days after the birth of the young animal. Colostrum is very high in albumin and consequently in total solids, but is somewhat deficient in lactose (milk sugar) as shown in the following analyses of colostrum from twenty cows:

#### AVERAGE COMPOSITION OF COLOSTRUM (ENGLING)

Fat	3.37
Casein	4.83
Albumin	15.85
Ash	1.78
Lactose	2.48
Total solids	28.31
Water	71.69
	100.00

Milk also varies in composition according to the period of lactation, the percentage of fat increasing toward the end of the period. "Strippings" are also richer in fat than "fore milk" or that drawn first from the udder.

Commercial Value of the Constituents of Milk. While in meat the proteins are the most expensive constituents, the fat being less highly prized and often wasted, in the case of milk the reverse is true, the commercial value being largely determined by the amount of fat present. In the form of butter, milk fat is worth two or three times as much as other animal fats, while skim milk, which differs from whole milk only in that the fat is largely removed, is a proverbially cheap food. Because of the high commercial value of the fat the determination of this constituent is the most important of the analytical processes which have been devised and in the buying and selling of milk is ordinarily the only one undertaken.

Milk Standards. In order to prevent the watering and skimming of market milk, as well as to exclude the product unduly poor in composition due to breed, individual characteristics, and other causes, standards have been fixed by Federal,

State and municipal authorities. The Federal standard, which has been adopted by various States and cities, excludes milk drawn fifteen days before and ten days after calving and requires not less than 8.5 per cent of solids not fat and not less than 3.25 per cent of milk fat.

Sampling of Milk. Proper sampling is very simple but too often neglected. An analysis of a sample taken from the bottom or the top of milk that has stood long enough for the cream to rise is worse than useless. The milk first drawn from the udder, like skim milk, is poor in fat, while the last of the milking is really cream. Whether the lot of milk be large or small, it should be well mixed before sampling. This is accomplished by thorough stirring with a dipper, by pouring three times from one pail or bottle to another, or, if the quantity is small, by shaking

in a bottle. Immediately before the sample is divided or portions are removed for analysis this mixing must be repeated.

Composite Samples. It is obviously impracticable to mix together the contents of several cans and still more so of all the cans of a large shipment. In such cases a composite sample accurately representing the whole lot may be secured by mixing small portions obtained from each can after thorough stirring. If the cans all contain approximately the same amount the portions can be of the same size, otherwise to be strictly accurate they should be proportional to the amount. This latter end is secured without calculation by using a sampling tube or "milk thief," which takes out a column equal in height to the height of the milk in the can.

The Scovell sampler, shown in Fig. 1, has holes in a cap at the bottom end which should be opened by pushing down before using. The tube is then slowly lowered to the bottom of the can,



Fig. 1.—Scovell Milk-sampling Tube.

allowing the milk which enters through the holes to rise to the same level as outside. The holes are closed by pushing the cap against the bottom of the can and the milk is delivered into the sample bottle.

A composite sample of the milk furnished from day to day may be secured in the same manner, adding a small amount of

potassium bichromate as a preservative. Such a sample may be tested at the end of a week or even a month.

\*Material for Laboratory Practice. For the analytical work, a quart of milk, a half pint of cream (not over 25 per cent fat), and a pint of skim milk should be provided. To the sample of milk add 2 to 3 drops of 40 per cent formaldehyde solution so that 1 part of the gas will be present in about 20,000 parts of the cream. To the sample of skim milk add 1 gram of borax. These preservatives will be tested for by well-known methods; they will not interfere with the quantitative determinations undertaken.

\*Determination of Specific Gravity of Milk by the Lactometer. Thoroughly mix the sample of whole milk as described. Transfer to a glass cylinder, insert a Quevenne lactometer (Fig. 2), and after the temperature becomes constant let each student read the density on the Quevenne scale and the temperature on the Fahrenheit scale. Correct to 60° F., using the table on page 211.

Fig. 2.—Quevenne Lactometer. The readings on the Quevenne scale are the figures in the hundredth and thousandth place of the specific gravity expressed as whole num-

bers. For example the reading 31 corresponds to the specific gravity 1.031. Accordingly to convert Quevenne readings into specific gravity prefix 1.0.

After determining the specific gravity again mix the whole milk sample and transfer to as many two-ounce bottles as there

\* See page o for explanation of the use of this star.

are students. Stopper each bottle with a clean cork. As only fat by the Babcock method will be determined in the cream and skim milk samples these need not be divided.

The specific gravity of milk of standard quality, which ranges between 1.02 and 1.035, is lowered by watering and raised by skimming. While the lactometer may detect one or the other fraud, unfortunately it may show a normal reading if both forms of adulteration have been practiced. It is accordingly necessary to determine, in addition to the specific gravity, the fat and calculate the total solids or else the total solids and calculate the fat. A more certain procedure is to determine both the fat and total solids by analysis, using the calculated amounts as a check.

Duplicate Determinations. The maxim "Eine Analyse ist Keine Analyse" should ever be kept in mind. No analyst, however experienced, is infallible. Even the agreement of the results by the same analyst is no proof of their accuracy, as the same error may have been made in both cases. It is therefore desirable in important work that different chemists make the determinations, using different reagents or even different methods.

The author strongly recommends that all analyses, except such few as are specially noted, be carried out by the student in duplicate. If two methods are used for the same constituent, as is true of the total solids and fat of milk, one determination by each method will suffice.

\*Determination of Total Solids of Milk in an Open Dish.

The dish should be of thin metal, with a flat bottom. If made

of platinum the ash can be determined after weighing the solids in the same dish by heating at dull redness in a muffle furnace. Owing to the expense of platinum it is recommended to use tinned lead dishes,  $2\frac{1}{2}$  in. in



Fig. 3.—Tinned Lead Dish.

diameter and  $1\frac{1}{16}$  in. high, such as are made for capping wide-mouth bottles; these cost less than two cents each and can be thrown away after using (Fig. 3). Aluminum or nickel dishes are suitable but cost more. Tin box covers answer the purpose.

Weigh accurately the dish—the use of a desiccator at this stage is unnecessary—mix the sample by shaking, and by means of a 5 cc. pipette transfer to the dish 5 grams, which will be slightly less than the contents of the pipette filled to the mark. Although the results are equally as good if any amount from 4 to 6 grams is used, still in practical work a great amount of figuring and possible mathematical error will be avoided by using exactly 5 grams, and with little additional labor.

An error of two or three milligrams in weighing the milk will not appreciably affect the result—in fact evaporation, if too long a time is taken up in the weighing, will cause a more serious error.

Evaporate on a boiling water bath, using a ring with an opening only slightly smaller than the bottom of the dish. At the end of two to three hours wipe the bottom of the dish dry, place



Fig. 4.—Desiccator with Wire Gauze Disk.

in a desiccator while hot, cool fifteen minutes, and weigh. Calculate the per cent of solids from this weight.

The desiccator for food analysis (Fig. 4), should be of good size (inside diameter at least 6 in.), so as to hold several dishes with a diameter of  $2\frac{1}{2}$  in. These can be supported on a circular piece of wire gauze, cut to fit the desiccator, or on a perforated porcelain plate. Three dishes can be arranged in a triangle and one placed in the middle on top of these. While the

evaporation is going on proceed according to the following method.

\*Determination of Total Solids of Milk by the Asbestos Method. This method, devised by Babcock, is really preliminary to the extraction of the fat with ether (p. 21). The determination of total solids is accordingly incidental and furnishes for our purpose a check on the open-dish method. When the percentage of fat is obtained by the Babcock centrifugal method,

the open-dish method furnishes the readiest means of determining the total solids.

Process. Heat for a minute or two in the flame of a Bunsen burner 2 to 2.5 grams of woolly asbestos (free from fine and brittle material), and introduce into a hollow cylinder of perforated sheet brass 60 mm. high and 20 mm. in diameter, closed 5 mm. from the bottom with a disk of the same material

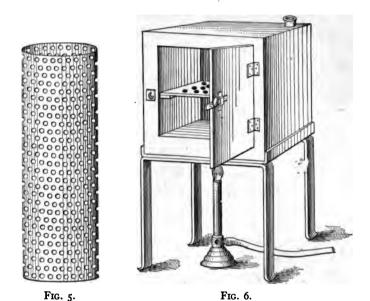


Fig. 5.—Perforated Metal Cylinder for Milk Analysis.

Fig. 6.—Water Oven.

(Fig. 5). The perforations should be 0.7 mm. in diameter and about 0.7 mm. apart. Cool in a desiccator and weigh. Shake the whole milk sample, measure out 5 cc. with a pipette, allow to deliver slowly on to the asbestos in the cylinder and weigh. As there is no means of removing any of the milk after it has been added to the asbestos, it is easier to use 5 cc. than exactly 5 grams.

Dry in a boiling water or steam oven (Fig. 6), for about four hours, cool in a desiccator, and weigh. During the first part.

of the drying, the door of the oven should be opened from time to time to allow escape of the water vapor. Half of the drying can be carried out on the day the portion is weighed out, the remainder on the next day before extracting with ether.

While the milk in the open dish and in the perforated cylinder is drying make single determinations of fat in the whole milk, skim milk, and cream by the Babcock test.

\* Determination of Fat of Whole Milk, Skim Milk, and

Cream by the Babcock Centrifugal Method ("Babcock Test"). The apparatus consists of a 17.6 cc. pipette for measuring the milk (Fig. 7), test bottles with different diameter of neck for milk (Fig. 8), skim milk (Fig. 9), and cream (Fig. 10), a cylinder of 17.5 cc. capacity for measuring the acid (Fig. 11), and a centrifugal machine. The skim-milk bottle has a double neck consisting of a larger tube for introducing the milk, acid, and water, and a smaller tube for measuring the fat. The centrifugal machine may be obtained in different sizes holding from 2 to 40 test bottles, and arranged for hand, steam, or electric power. A 12-bottle hand machine (Fig. 12), answers very well for laboratory use.

Process. Mix the samples and pipette 17.6 cc. (whole milk and skim milk), or weigh 18 grams (cream) into the appropriate test bottle. The pipette should be rinsed with a few cc. of the milk to be tested before measuring out the portion for analysis. The pipetted portions of the whole milk and skim milk will weigh sufficiently near 18 grams for practical purposes. Cream, however, varies greatly in specific gravity according to its thickness, furthermore it froths and

Fig. 7.—Babcock Milk Pipette.

clings to the sides of the pipette. In order to insure accurate results the test bottle should be weighed before introducing the cream and enough cream added to increase its weight 18

grams. The chemical balance may be used, but weighing closer than 0.05 gram (about one drop) is unnecessary.

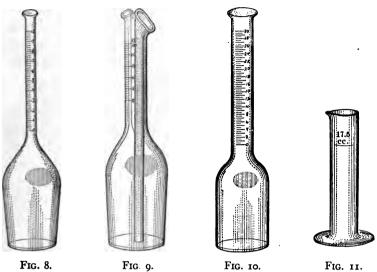


Fig. 8.—Babcock Milk Test Bottle.
Fig. 9.—Wagner Skim Milk Test Bottle.
Fig. 10.—Winton Cream Test Bottle.
Fig. 11.—Babcock Acid Measure.

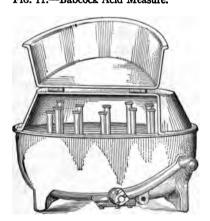


Fig. 12.—Babcock Centrifuge.

Introduce 17.5 cc. of commercial sulphuric acid (sp.gr. 1.82 to 1.84).

In the sample containing formaldehyde note that a violet color appears at the juncture of the two liquids, whereas in the other samples only a dirty brown color is evident. The violet color is dependent on the presence of iron salts in the commercial acid. The same color is obtained if a portion of the milk containing formaldehyde is heated with an equal volume of of concentrated hydrochloric acid containing in one liter 2 cc. of 10 per cent ferric chloride solution.

Immediately after adding the acid mix the milk and acid thoroughly by a vigorous rotatory motion, holding the test bottle

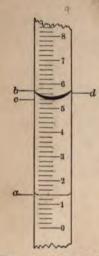


Fig. 13.—Neck of Milk Test Bottle, Showing Top and Bottom of Fat Column.

by the neck at a slight angle away from the body. Much heat is developed and the lumps of curd, which at first form, gradually disappear on shaking. After shaking each bottle place in a pocket of the centrifugal machine. If all the pockets are not used arrange the bottles symmetrically to avoid excessive vibration. If the machine is cold it should be heated by a quart or more of boiling water. When the machine is full, whirl at the rate of 800 to 1000 revolutions per minute, according to the diameter of the frame, for five minutes. Fill each bottle to the neck with boiling water from a wash bottle and whirl two minutes longer. Add boiling water nearly up to the top graduation, whirl again for two minutes, and immerse the bottles nearly to the top of the neck in a tank of water at about 60° C.

Remove one at a time for reading the fat column. Read the top of the top meniscus (Fig. 13, b) and immediately after the bottom of the bottom meniscus (Fig. 13, a). The difference between the two readings is the percentage of fat.

Empty the bottles while hot, shaking continually, and clean with hot water.

Although both the milk and the fat are measured the results are in percentage by weight. As already stated 17.6 cc. of milk

weigh approximately 18 grams. The volume corresponding to 10 per cent of fat on the neck of the test bottle is 2 cc. As the specific gravity of the liquid fat is 0.9, 2 cc. corresponds to 1.8 grams of fat and, therefore, to 10 per cent of 18 grams of milk.

After the milk or cream is pipetted into the test bottle the remainder of the process may be postponed a day or two, as souring does not affect the results. When these tests are finished the total solids by the open-dish method can be cooled in a desiccator and weighed.

\*Determination of Fat by Extraction with Ether. On the next day, while the drying of the milk by the asbestos method is being finished, preparations should be made for the extraction of the fat with ether.

Ether extraction, whether of fat in milk or of the crude fat in animal or vegetable products, is carried out in a so-called continuous extractor, i.e., an apparatus in which the ether, after dissolving a portion of the fat of the material and discharging into the extraction flask, is volatilized, condensed, and again allowed to act on the material, the steps in the process being repeated automatically and continuously until the extraction is complete.

The Soxhlet Extractor, shown in Fig. 14, depends on the intermittent action of a glass syphon. The ether gradually condenses into the extraction tube containing the material until it rises to the top of the siphon, when it is discharged into the extraction flask. This ingenious apparatus, although well adapted for certain purposes, is not thoroughly satisfactory for the determination of fat or ether extract, as it is fragile, expensive, employs a large quantity of ether, and requires too large an extraction flask for accurate weighing.

The Johnson Extractor 1 (Fig. 15) obviates all these defects.

The extractor proper E consists of a vertical tube 175 mm. long and 26 mm. in diameter (inside measurement), provided with a bulge at the bottom, to prevent trapping of the condensed ether, and a delivery tube attached at an angle of about 45°, so that the condensed ether strikes the neck of the extraction flask

<sup>&</sup>lt;sup>1</sup> S. W. Johnson, Amer. Jour. Sci., 1877, p. 196.

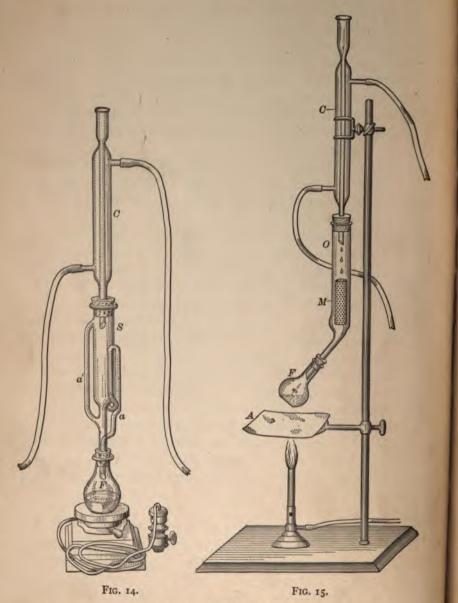


Fig. 14.—Soxhlet Extractor.

Fig. 15.—Johnson Fat Extractor with Perforated Cylinder for Milk Analysis.

F, thus avoiding spattering. The extraction flask has a capacity of 30 to 35 cc. and is attached to the delivery tube by a carefully bored cork. The reflux condenser C is merely an ordinary Liebig condenser, set up in a vertical position and attached by a bored cork at its delivery end to the extractor. The same condenser can be used both for distillation and refluxing, according to the way it is set up. A support with a suitable

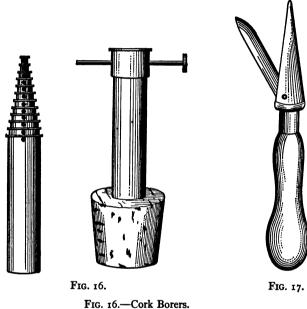


Fig. 16.—Cork Borers.
Fig. 17.—Cork Borer Sharpener.

clamp serves to hold the condenser firmly in position; the extractor and extraction flask hang suspended with no support other than the corks by which they are attached. The flask is heated by a Bunsen flame impinging against a piece of sheet metal which rests on a ring attached to the condenser support.

Instructions. Although only a single extraction of fat in milk need be made, the duplicate determination having been carried out by the Babcock centrifugal method, two extractors connected with Liebig condensers should be set up, as both will

be needed later in the determination of the crude fat in vegetable products. In boring holes in the corks (which should first be rolled until soft), be sure that the borer (Fig. 16) has a keen edge, secured by means of a "cork borer sharpener" (Fig. 17), and that it bores a smooth hole into which the tube fits without danger of leaking. Do not use a rat-tail file; it will be found that one of the borers in a good set, properly sharpened, will cut a hole into which, without further treatment, a given tube will fit accurately

When the apparatus is set up, place an identification mark on the extraction flask. This is best done with a lead pencil on an etched or ground spot. The etching fluid, known as "diamond ink," applied with a brush, or a few strokes with the flat surface of a file moistened with water, gives the desired surface. In using any hydrofluoric acid preparation be careful not to get any on the skin, as it makes serious wounds.

Weigh the flask without drying in a desiccator, and without a stopper. If special accuracy were important a counterpoise flask of the same size but slightly less weight could be used, thus obviating the slight error due to the variable amount of moisture which condenses on the surface.

Attach the flask to the lower end of the extractor and place the dried and weighed perforated cylinder with asbestos and milk solids in the extractor. Pour 8 to 10 cc. of anhydrous alcohol-free ether through the cylinder, attach the extractor to the condenser, run water through the latter, and heat the flask cautiously. Ether free from water and alcohol is required, as these would extract sugars and other substances from the residue. The ether in the form of vapor passes up through the extractor, is liquefied in the condenser, and is returned drop by drop through the asbestos into the extraction flask. The fat gradually extracted from the milk solids remains in the flask, but the vaporization and condensation of the ether continue without intermission as long as the heat is applied.

After two hours—the end of the laboratory period—the extraction is complete. Turn out the lamps, remove the flask, and allow to stand until the next day, when the ether should be

driven off over a register or in some other warm place and the flask, with the fat, dried in the boiling water oven for two hours. Cool, weigh, and calculate the percentage of fat. Compare with the percentage obtained by the Babcock test, also compare the percentages of solids obtained by the two methods.

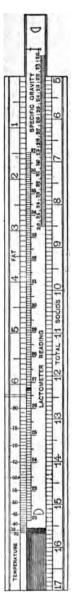
The percentages of fat by the extraction method are accurate to the second place of decimals, while those by the Babcock test vary from one- to two-tenths of a per cent. For ordinary purposes the shorter method is sufficiently accurate. Practically all the milk and cream sold for butter and cheese making in the United States are now valued by the Babcock test.

\*Calculation of the Total Solids from the Specific Gravity and Fat. Given these data a close approximation to the true percentage of total solids may be obtained from the table on page 212 or by the use of the Richmond slide rule (Fig. 18). Compare the results thus secured with those by direct drying.

\*Testing Milk for Borax and Boric Acid. Test the whole milk and skim milk by the following method: To 10 cc. of the sample in a watch-glass, add 6 drops of concentrated hydrochloric acid and mix thoroughly with a glass rod. Moisten a strip of turmeric paper with the mixture and dry on a clean watch-glass heated over a water bath. If borax or boric acid is present the paper will turn brick red, changing to a greenish color with a drop of ammonia water.

Brief Statements of Methods for the Deter- Fig. 18.—Richmond mination of Other Constituents of Milk. The Milk Scale.

Protein, including casein and albumin, is obtained by determin-



ing the total nitrogen by the Kjeldahl method (p. 65), and multiplying by the factor 6.38. Lactose or milk sugar is estimated by copper reduction, following the same method as is used in the estimation of dextrose (p. 76), but taking into account the difference in the reducing power of two sugars. Before applying the method it is necessary to remove the proteins and fat by precipitation with copper sulphate. The proteins form copper compounds which entangle the fat mechanically, thus permitting the removal of both classes of interfering substances by one filtration.

#### BUTTER

Composition of Butter. Butter consists of the fat of milk mechanically mixed with water, a small amount of casein or curd, and added salt. Traces of lactic acid resulting from the fermentation of the sugar are also present. The average composition of 350 samples analyzed by Farrington 1 at the Chicago World's Fair, is as follows:

Water	11.57
Fat	84.70
Curd (casein)	-95
Ash (including salt)	2.78
	100.00

\*Preparation of Sample of Butter for Analysis. Place a half pound of butter in a pint fruit jar, fasten the cover securely in place, and keep in a warm place or in hot water until the butter is melted. As lumps of the butter may remain unmelted for some time, care should be taken to heat long enough to melt completely the whole mass. Without opening, cool the jar and contents under a stream of cold water, shaking continually. When the mass of butter has solidified, dry off the outside of the jar and keep in a refrigerator until needed. The sample thus prepared (previous to the exercise) will be homogeneous and sufficient for the duplicate analyses of over fifty students.

<sup>1</sup> Farrington and Woll: Testing Milk and its Products, 23d ed., p. 259.

BUTTER 27

\*Determination of Water, Fat, Curd, and Ash of Butter in One Weighed Portion. Weigh two tinned lead dishes, such as were used for the determination of total solids in milk (p. 15), and place in each dish 2 grams of the butter sample. Dry in a boiling water-oven for two and one-half hours, cool in a desiccator, and calculate the loss in weight as percentage of moisture.

While the dishes are in the oven prepare two porcelain Gooch

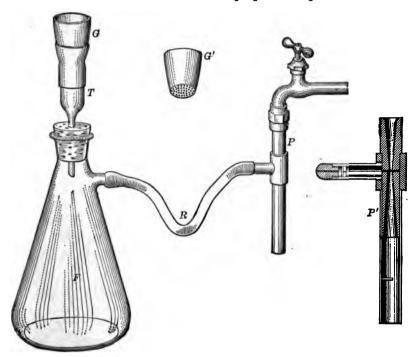


Fig. 19.—Filtering Apparatus for Gooch Crucibles with Chapman Pump.

crucibles, diameter 35 mm. (Fig. 19 G'), as follows: Connect the crucible G by means of a piece of large, thick rubber tubing with the filter tube T, the stem of which passes through the rubber cork of the tubulated Erlenmeyer flask F, made of thick glass so as to resist a vacuum. Connect the tubulature with the filter pump P and pour on the crucible a quantity of pulped asbestos, suspended in water, sufficient to form a blanket about  $\frac{1}{4}$  in. thick.

The asbestos used should have previously been chopped into small pieces, digested with hydrochloric acid (sp.gr. 1.125) on a water bath for an hour or two, and washed by decantation. When needed it is shaken with water and removed to the crucible while in suspension, using suction. Asbestos prepared for filtering copper suboxide in sugar analysis may also be used (p. 76).

Wash once with water and, to facilitate drying, with a little alcohol. Dry cautiously over a piece of asbestos paper, finally raising the heat to a scorching temperature. Allow to cool, at first in the air, finally in a desiccator and weigh.

To each of the dried residues obtained in the water determination add enough gasoline from a wash bottle to about half fill the dish and stir carefully with a short glass rod. By means of the rod form a lip on the edge of the dish. Pour the gasolene and any suspended matter onto one of the Gooch crucibles connected with the suction apparatus. Repeat the treatment several times until the fat appears to have been dissolved, then transfer to the crucible all the insoluble matter, using a "policeman," or the ball of the little finger, and a stream of gasolene to remove any that may adhere to the dish. When the dish is clean, wash down the sides of the Gooch crucible with gasolene and continue the washing with several more portions, allowing the crucible to empty after each addition.

Dry the crucible in a boiling water oven for one to two hours, cool in a desiccator and weigh. The increase in weight is ash (including salt) and curd.

Ignite cautiously on a piece of asbestos paper, or in a muffle furnace, at a dull red heat until the residue is white or gray. Cool (finally in a desiccator) and weigh. The loss since the preceding weighing is curd (casein), the difference between the final weight and the weight of the crucible as first prepared is ash, including salt. Calculate both curd and ash in percentages of the butter sample.

The characters of butter fat, as compared with other fats, will be considered in Chapter VII.

The Gooch Crucible used in the preceding and many other methods of analysis is a great labor saver in the analytical CHEESE 29

laboratory. Before its invention it was customary to perform all filtrations on filter paper, which not only required more time, but necessitated drying of the paper with its contents at 100° C., or else, when the nature of the precipitate permitted, igniting in a crucible of the ordinary type until the paper was destroyed. In the latter case a correction for the ash of the filter was necessary. The Gooch crucible is really a combination of a filter and a crucible. It may be obtained made of either platinum or porcelain.

#### CHEESE

Composition of Cheese. Cheese is prepared by the action of rennet (a preparation from calf's stomach) on milk. The casein is coagulated and the fat is mechanically held by the casein, while the whey, containing the sugar, albumin, and certain ash constituents, is drained off. The cheese is finally salted, pressed, and cured. The numerous varieties of cheese owe their characteristics to the kind of milk used (cow's, sheep's, goat's, etc.), the process of manufacture, and the nature of the organisms introduced. The following table of analyses taken from Doane and Lawson's compilation, shows the composition of common European and American cheese:

### COMPOSITION OF CHEESE

	Analyst.	Number of Samples.	Water.	Fat.	Casein, etc.	Lactose and Lactic Acid.	Ash, including Salt.
· Brie	Balland	I	48.80	22.45	19.94	4.85	3.9 <b>6</b>
Camembert	Balland	I			18.72		4.68
Cheddar (American)	Van Slyke	9		ı	24.45	1	
Edam	Patrick	I	-		28.41		5 · 5 <b>5</b>
Gorgonzola	Musso	7	37.30	34.67	25.16	1.62	3.82
Limburg (American)	Winton	1	42.12	29.40	23.00	0.38	5.10
Neufchatel (American)	Winton	1	57.25	22.30	15.03	2.94	2.48
Pineapple (American)	Winton	4	24.07	38.12	29.35	2.49	5.69
Roquefort	Winton	I	39.28	29.53	22.62	1.77	6.80
Swiss (Emmental)	Benecke	7	37.77	23.92	30.97		6.85

<sup>1</sup> U. S. Dept. Agr., Bur. Animal Industry, Bul. 146.

Analysis of Cheese. Although laboratory work in the analysis of cheese seems beyond the province of this book, a brief consideration of the analytical methods should be given. Water is determined by drying in an open dish as in the case of milk (p. 15) and butter, but the time required is longer. Protein is calculated from the nitrogen, as determined by the Kjeldahl method, using the factor 6.38. Fat may be determined by a modification of the Babcock test or more accurately by ether extraction. In the latter case the cheese, as first proposed by Short, is ground up in a mortar with anhydrous copper sulphate which is converted into the ordinary or hydrous form by the absorption of the water of the cheese, thus obviating the necessity of drying. Ash, including salt, is obtained by heating below redness.

### OTHER DAIRY PRODUCTS

Condensed Milk. Milk concentrated to about half its original volume is known as evaporated milk when nothing is added, and as sweetened condensed milk when mixed with sugar. Ordinarily both products are sterilized and put up in hermetically sealed tin cans.

The Methods of Analysis for the unsweetened product are the same as are used for milk, allowing for the greater concentration.

The presence of sucrose in sweetened condensed milk necessitates the use of special methods for the determination of fat and sugars. The amount of Fat is best found by the Roese-Gottlieb method, which consists in shaking the milk, after adding ammonia water, with a mixture of alcohol, ether, and petroleum ether, separating the solvent layer, and evaporating. Sucrose is obtained by difference, subtracting the results of direct determinations of water, fat, protein, ash, and lactose from 100.

Ice Cream. In addition to milk, cream, and flavors, ice cream often contains thickeners, such as gelatin and starch, artificial colors, and sometimes chemical preservatives. Homo-

<sup>1</sup> Land. Vers. 1892, 40.

genized (emulsified) foreign oils may be substituted for part of the butter fat.

Methods of Analysis. The Roese-Gottlieb method is suitable for determining the Fat, which is the most important constituent. Homogenized Oils are detected by separating the fat by Paul's method, saponifying by the Leffmann and Beam method, and distilling the volatile fatty acids. Thickeners are tested for by Patrick's method.<sup>2</sup> Preservatives and Colors are found by the usual tests.

<sup>1</sup> U. S. Dept. Agr. Bur. Chem. Bul. 162, p. 118. <sup>2</sup> Ibid. Bul. 116, p. 26.

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# CHAPTER III

## MEAT AND FISH

Chief Constituents. Lean meat consists essentially of Muscle Fibers, Connective Tissues, and Fat Cells. The fibers of ordinary meat (Fig. 20) are striated and have a sheath, known

as the Sarcolemma, made up chiefly of a protein related to elastin, insoluble in ordinary neutral reagents and belonging therefore to the albuminoids. Within the sarcolemma is contained the meat juice containing several proteins, of which Myosin is the most important.

Elastin and Collagen are the chief constituents of connective tissue. Gelatin is derived from collagen by boiling with water and is often classed with the albuminoids.

Glycogen, a carbohydrate closely related to starch and dextrin, is present in large amounts in liver and in small quantities in meat and fish muscle. Other sugars occur in minute quantities.

Xanthine Bodies, or purin bases (Xanthine, Carnine, Guanine, etc.) closely related to the obromine of chocolate and caffeine of tea and coffee, Creatine, Creatinine, and other extractive substances, are also present in meat and are characteristic constituents of meat extracts.

Fat occurs not only in the fat proper, but also in tens distributed throughout the lean portion of the meat.

Mineral Constituents, such as are contained in milk, are also present.

From these brief statements, it is evident that meat is far

from a simple substance. Further consideration of the individual chemical substances would take us within the realm of physiological chemistry, where we would find much unexplored territory. For our purpose it is chiefly desirable to consider the groups of nutritive substances present. As in the case of milk, these groups are: (1) protein, (2) fat, (3) ash, and (4) carbohydrates. It should be emphasized, however, that while milk contains a carbohydrate, lactose, as one of its chief constituents, animal muscle contains such a small amount of carbohydrate matter that it can, for ordinary purposes, be ignored.

Composition of Meat, Fish, and Eggs. The table on page 35 gives the average composition and fuel values of some of the cuts of beef, veal, mutton, and pork, fowls, certain species of fish and shellfish, and eggs.

Analysis of Meats. The meat must first be separated into lean, visible fat, and bone, and the percentage of each determined. The samples of lean meat and visible fat thus obtained are then separately analyzed. Suitable methods have been published by Grindley and Emmett.1 The solids may be determined by drying at 100° C., as in the case of milk, but more accurately by the desiccator method, the fat by ether extraction or a centrifugal method, the ash by burning at dull redness, and the protein by calculation from the nitrogen. The methods, however, do not yield such accurate results as in the analysis of milk, owing to the greater difficulties in sampling, the more complex composition, and other causes. It is particularly difficult to obtain the full amount of fat by ether extraction even after long treatment and repeated grinding. As for the determination of the minor constituents, the methods are often complicated and suited only for special investigations.

f Meat Extracts. Micko<sup>2</sup> has devised methods for es a general the amounts of the extractive substances noted in the preceding paragraphs. Bigelow and 6 k<sup>3</sup> and Street 4

<sup>&</sup>lt;sup>1</sup> Jour. Amer. Chem. Soc., 1904, 27, 658; 1905, 28, 25.

Ztschr. Unters. Nahr. Genussm., 1902 et seq.
 U. S. Dept. Agr. Bur. Chem., Bul. 114.
 Conn. Agrl. Expt. Sta. Rep., 1908, p. 606.

COMPOSITION

Average Composition of Meat, Fish, and Eggs (Atwater and Bryant  $^{1}$ )

	Refuse.	Water.	Fat.	Protein (N X 64).	ı, Ash.	Carbo- hydrates.	Calories per Pound.
Meat:							
Beef, ribs	20. I	45.3	20.0	14.4	0.7		1110
Sirloin steak	12.8	.54.0	16.1	16.5	0.9		985
Porterhouse steak	12.7	52.4	17.9	19.1	0.8		1110
Round steak	8.5	62.5	9.2	19.2	1.0		745
Rump	19.0	46.9	18.6	15.2	0.8		1065
Beef liver	7.3	65.6	3.1	20.2	1.3	2.5	555
Corned beef, canned	0.0	51.8	18.7	26.3	4.0		1280
Veal cutlet	3.4	68.3	7.5	20. I	1.0		690
Mutton, leg	17.7	51.9	14.5	15.4	0.8		900
Mutton, loin	14.8	40.4	31.5	13.1	0.6		1575
Pork, ribs	18.1	41.8	25.5	14.1	0.8		1340
Ham, smoked	12.2	35.8	33.2	14.5	4.2		1670
Bacon, smoked	8.7	18.4	59.4	9.5	4 · 5		2685
Pork, salt, fat	0.0	7.9	86.2	1.9	3.9		3670
Fowls	25.9	47.1	12.3	13.7	0.7		775
Fish:							ļ
Bluefish, dressed	48.6	40.3	0.6	10.0	0.7		210
Eels, dressed	20.2	57.2	7.2	14.8	0.8		580
Shad	50.1	35.2	4.8	9.4	0.7		380
Shad roe	0.0	71.2	3.8	20.9	1.5	2.6	600
Halibut steak	17.7	61.9	4.4	15.3	0.9		470
White fish	53 · 5	32.5	3.0	10.6	0.7		325
Trout, brook	48.1	40.4	1.1	9.9	0.6		230
Salmon, canned	14.2	56.8	7.5	19.5	2.0		68ơ
Codfish, salt, bone-		ĺ				ļ	
less	1.6	54.8	0.3	27.7	14.7		545
Shellfish:		ĺ				<i>.</i>	l
Lobster, whole	61.7	30.7	0.7	5.9	0.8		140
Oysters, meats	0.0	88.3	1.3	6.0	1.1		230
Scallops, meats	0.0	80.3	0.1	14.8	1.4	3.4	345
Eggs:		[					
Hen's	11.2	65.5	9.3	11.9	0.9		635

<sup>&</sup>lt;sup>1</sup> The Chemical Composition of American Food Materials.

have made extensive investigations of the meat extracts on the market in the United States.

Food Preservatives. Toward the end of the nineteenth century the attention of the public was directed to the preservation of foods with borax, boric acid, salicylic acid, sulphurous acid, sulphites, and fluorides. Although certain of these had been on the market as household preservatives, their extensive use by manufacturers in sausage, Hamburg steak, oysters, and other meat and fish foods, also in catsups, preserves, jellies, fruit juices, syrups, and dried fruits was not generally known until after the passage of State food laws and the publication of State reports.

In order to settle the controversies which arose between food officials and food manufacturers, physiological experiments were undertaken by government chemists, members of a board of consulting scientists appointed by the President, and scientists representing certain trade interests. The results obtained were conflicting, but the findings of the Board were accepted by the Secretary of Agriculture as final, and as a consequence all the preservatives but sodium benzoate, which had largely taken the place of salicylic acid, and sulphurous acid, were excluded from foods, and these two were allowed only in limited amount and with a declaration on the label. Exception was made in the case of foods such as dried codfish, from which the preservative could be removed by soaking.

The Federal decisions were not acceptable to all scientists or manufacturers, and as a consequence, bitter controversies arose, food officials, physicians, physiological chemists, manufacturers, and publicists aligning themselves on one side or the other. While the manufacturers using preservatives complied with the requirement of declaring the presence of the preservative in a certain size type, those not using it often declared its absence in much larger type. Prominent associations also came out against the use of chemical preservatives.

The principal arguments in favor of the conservative use of chemical preservatives are that they prevent the development of dangerous germs, that they permit the marketing of foods in a sweet and sound condition which might reach the table fermented or otherwise spoiled, that they cheapen the cost of foods to the consumer, that they take the place of highly flavored and unwholesome products, such as spices, wood smoke, etc., and that in the quantities permitted they are not injurious to health—benzoic acid, for example, being readily eliminated from the body as hippuric acid.

The arguments against the use of preservatives are that they permit the marketing of spoiled food whether or not containing dangerous germs, that they enrich the producer at the expense of the consumer, that they are not, like spices and other time-honored preservatives, evident by their taste or odor, which of themselves are desirable, and that they are injurious to health partly by preventing the proper digestion of foods, owing to their antiseptic action, and partly because of their toxic nature.

Without going further into the controversy it may be stated that the determination of benzoic acid and sulphurous acid, either free or combined, often demands the attention of the food chemist. Sodium benzoate is more commonly used in fruit products, such as catsup, jam, and soda water syrup, sulphurous acid in dried fruits, and sodium or calcium sulphite or bisulphite in meat and fish products. Borax and boric acid are still used in dried codfish, but as this food is soaked in water before cooking, thus removing the preservative, the practice comes within a special provision of most food laws.

The use of sulphites in Hamburg steak and sausage is objectionable if for no other reason because it permits the marketing of decomposed meat, serving not merely as a preservative but as a deodorizer.

Salt, wood smoke, vinegar, spices, and sugar have been used as preservatives in meat and fish products, as well as other foods, from time immemorial, and they have served in many cases where refrigeration, desiccation, and sterilization have not been available; furthermore, they all impart desirable flavors and two of them—salt and sugar—are valuable elements of nutrition.

\*Determination of Sulphur Dioxide in Hamburg Steak. Material for Laboratory Practice. To 1000 grams of chopped round steak add 2 grams of sodium sulphite. Mix thoroughly by kneading.

Apparatus (Fig. 21). S is a 500-cc. distilling flask in which the substance is placed. It is provided with a double-bored rubber stopper through which pass on one side the delivery tube



Fig. 21.—Apparatus for Determination of Sulphur Dioxide.

from a carbon dioxide apparatus reaching nearly to the liquid in the flask, and on the other a bulb tube B, connected with an upright condenser C, both of the latter being the same as used for the Reichert-Meissl and Polenske methods (p. 158). Attached to the lower end of the condenser, by means of a piece of rubber tubing, is a glass tube of such a length that it reaches nearly to the bottom of the receiving flask R.

The carbon dioxide is generated by the action of dilute hydro-

chloric acid (sp.gr. 1.125), delivered from a 125-cc. separatory funnel F, on lumps of marble contained in a 250-cc. salt-mouthed bottle M. The gas is freed from any possible contamination of sulphur dioxide by passing through a wash bottle consisting of a 125-cc. salt-mouthed bottle W containing a dilute sodium hydroxide solution. The bent tube connecting the two bottles passes just through the stopper of M, but nearly to the bottom of W. The delivery tube in S passes about half way to the bottom, so that it does not dip below the liquid when the flask is half filled.

Process. Place 50 grams of the mixture of meat and sulphite (weighed on a balance accurate to 0.5 gram) in the flask S, add 200 cc. of water, and introduce the stopper with connecting tubes. Place enough water in the receiving flask R so that the delivery tube dips below the surface and add bromine water sufficient to impart a distinct yellow color.

Partially open the stopcock of F and allow the acid to deliver drop by drop on the marble. The flow of carbon dioxide should be uniform and at a moderate rate, as shown by the escape of the bubbles through the liquid in W. After running a few minutes to insure the removal of all air, remove the stopper with tubes from S and without delay introduce from a pipette 5 cc. of a 20 per cent solution of phosphoric acid. The carbon dioxide gas, being heavier than air, does not escape from the flask. Close the flask immediately, bring to boiling with a Bunsen flame, and continue the boiling until the distillate measures about 150 cc. If the yellow color of the liquid in the receiver disappears, add more bromine, repeating if necessary. The carbon dioxide prevents oxidation of the sulphur dioxide which would take place in air.

Carry out a duplicate distillation, using the same apparatus, then proceed as follows:

After the distillation is complete boil the distillate until the excess of bromine is removed as shown by the odor, remove to a beaker, rinsing with water, and dilute further to about 250 cc. Add 1 cc. of concentrated hydrochloric acid, heat to boiling,

and add barium chloride solution drop by drop until the precipitate no longer forms. Allow to stand in a warm place overnight or longer.

Prepare a porcelain Gooch crucible with a compact mat of amphibole asbestos, about  $\frac{1}{4}$  in. thick, ignite at bright redness, cool, and weigh (see page 76).

After weighing the Gooch crucible place it again in the filtering apparatus, apply suction, decant the liquid from the precipitate of barium sulphate onto the crucible, and finally transfer the precipitate to the crucible by means of a stream of hot water from a wash bottle. Remove any adhering barium sulphate from the beaker, using a "policeman." Wash about five times, nearly filling the crucible each time and allowing one portion to run through before adding another. Dry at a low temperature on a piece of asbestos paper heated by a small Bunsen flame, raise the heat cautiously, and finally ignite at dull redness for three minutes. Cool in a desiccator and weigh.

Calculate the percentage of sulphur dioxide (SO<sub>2</sub>) in the material, using the following formula:

$$P = \frac{64.06 \times a \times 100}{233.43 \times 50} = 0.5489a,$$

in which P =the percentage of  $SO_2$ , 64.06 = 32.06 + 32 =the molecular weight of  $SO_2$ , 233.43 = 137.37 + 32.06 + 64 =the molecular weight of  $BaSO_4$ , and a =the weight of  $BaSO_4$  found.

### CHAPTER IV

### NATURAL VEGETABLE FOODS AND MILL PRODUCTS

Definition. Natural vegetable foods may be defined as products that reach the consumer exactly as they are taken from the plant, such as grain, seeds, vegetables, fruits, and nuts. The constituents present in these also occur in products of cereals, oil seeds, leguminous seeds, spices, coffee, cocoa, etc., obtained by grinding with or without other mechanical treatment that changes the amount, but not materially the kind, of the constituents, such as sifting to remove bran, pressing to remove a portion of the oil, or roasting to develop flavor.

The Six Groups of Constituents. Since natural vegetable products contain the substances essential for animal growth, namely proteins, fat, carbohydrates, and mineral salts, together with crude fiber and water as incidental constituents, determinations of the six groups of constituents taken up in Chapter I are of first importance. Starch, which makes up nearly all the nitrogen-free extract and about 75 per cent of the dry matter of the cereals, also sugars and pentosans, which occur widely distributed, are frequently determined, although for most purposes the amount of nitrogen-free extract answers the requirements.

The division into the six groups and the general scheme of analysis originated about the middle of the nineteenth century with the German agricultural chemists as a basis for feeding farm animals. As the general principles of human and animal nutrition are the same, analysis of foods for man and beast are made by the same methods and expressed in the same terms.

These same methods also serve for food inspection and commercial food analysis.

Criticisms of the Methods. Not one of the methods gives results which can be expressed in terms of definite composition such as is possible in inorganic analysis. Each of the six groups contains not only substances related in chemical composition, but also others related only in physical properties such as volatility or solubility, or in nitrogen content, as will be considered in connection with each method. The fact that the tables of composition of both human and animal foods and efficient systems of feeding are based on these methods is sufficient reason for their continuance.

Composition of Typical Products. The averages of analyses of a few common cereal, legume, and oil-seed products, used as food for man or cattle, appear in the table on page 44, and of vegetables, fruits, and nuts in the table on page 45. In addition to the percentages of the six groups of constituents, the fuel values in calories per pound, calculated as described on page 4, are also given. The materials selected are only a few of the hundreds on the market, but are sufficient to illustrate the range in composition. No analyses of bakers' products are included. Ordinary bread has practically the same composition as the flour from which it is made, allowing for water and small amounts of shortening, salt, and yeast added. Cake and pastries differ widely in composition, owing to the kind and proportion of the ingredients. The average composition of spices is given in the table on page 46, and of tea, coffee, and cocoa on pages 207, 204, and 209.

\*Material for Laboratory Practice. Any food in the table on p. 44, except peanut butter, which, owing to its pasty condition is difficult to handle, is suited for analysis by the student. Coffee and tea, analyses of which appear on pp. 204 and 207, may also be used, taking care to note that the nitrogen is partly from caffeine and therefore the factor 6.25 cannot be applied until a correction for the caffeine nitrogen has been introduced. Cocoa and chocolate, owing to their

slow filtrations, would better be avoided. Although the same methods apply to all these, the experience differs somewhat according to the nature of the product. It will add greatly to the interest if each student works on a different product, comparing at the end his analysis with those of his colleagues.

The constituents of vegetables, fruits, and nuts are determined by the same methods as are employed for cereal and allied products, but the preparation of samples of succulent vegetables and fruits and of oily nuts necessitates special treatment. It is therefore inadvisable for the student to attempt the analysis of any of these products except he can devote extra time to the work.

If the student is specially interested in spices and can devote a little extra time to his laboratory work, he may analyze one of the spices given in the table on p. 46, following certain modifications of the methods necessitated by the presence of piperine in black and white pepper and of volatile or essential oil in all the spices. The modifications required are noted after the descriptions of the methods for water, fat (ether extract), and protein. Ordinarily it will be found less confusing to confine the work to products other than spices.

The results obtained by the student in his analysis will differ somewhat from those given in the tables, as the products vary in composition within certain limits, dependent on the cultivated variety, locality of growth, season, and process of manufacture. It is this variation that makes analyses necessary.

Whatever the material selected, prepare the sample for analysis and make duplicate determinations of all the constituents given in the table by the methods described in detail in the following sections.

# 44 NATURAL VEGET WBLE FOODS AND MILL PRODUCTO

AVERAGE COMPC TION OF CEREAL, LEGUME, AND OIL-SEED PRODUCTS

	Water.	Fat (Ether Extract).	Crude Fiber.	Protein (N ×64).	Ash.	Nitrogen- free Extract.	Calories Per Pound.
Wheat flour 1	12.42	1.09	0.18	10.84	0.48	74.99	1646
Graham flour 1	13.09	1.71	1.87	11.67	1.77	69.89	1624
Rye flour 1	1	0.84	0.41	6.65	0.72	78.28	1622
Buckwheat flour 1	14.55	1.44	0.34	6.89	1.00	75.78	1605
Corn meal (unbolted) 1.	14.98	3.77	1.90	9.17	1.42	68.76	1643
Oat ineal 1	7.85	7.c6	0.86	14.66	2.01	67 . 5 <b>6</b>	1843
Rice 1	12.44	0.35	0.19	7.44	0.38	79.20	1630
Hominy (Grits) 1		0.44	0.32	8.25	0.38	77.12	1613
Cream of Wheat 2	10.20	I . 20	0.30	13.20	0.40	74.70	1692
Force 2		1.40	2.00	11.60	2.80	76.80	1740
Corn flakes3	10.33	0.31	0.48	6.50	2.36	80.02	1631
Grape Nuts 2		1.10	1.90	12.60	1.80	78.40	1773
Beans 1		1.62	3.20	20.37	2	r6.71	1728
Peanut butter 4		46.54	2.20	29.30	5	4.89	2829
Wheat bran 1	11.91	ું .૦૩	8.99	15.42	5.	53 . 87	1626
Rye bran 1	11.64	. 2.81	3.48	14.74	3 · 59	63 . 74	1643
Linseed meal, old pro.1	9.16	7.91	8.88	32.93	5.72	35.40	1770
Linseed meal, new pro.1	10.0	2.99	9.49	33.17	5.82	38. <b>46</b>	1562
Cotton seed meal 1	8.17	3.08	5.62	42.31	7.17	23.65	1883

<sup>&</sup>lt;sup>1</sup> Jenkins and Winton, Compilar Agriculture, Office Expt. Stations, 1

Analyses of American Feeding Stuffs, U.S. Dept.

<sup>&</sup>lt;sup>2</sup> Merrill and Mansfield, Cereal E ... Kfast Foods, Maine Agri. Expt. Station, Bul. 84. <sup>3</sup> Frear, Given, and Broomell, Breakfast Foods, Penn. Dept. Agr., Dairy and Food Div., Bul. 162, p. 14.

<sup>4</sup> Winton, Conn. Agri. Expt. Station., Rep. 1899, p. 138. (Ash includes 4.09% salt)

Average Composition of Vegetables, Fruits, and  $_{Ta}$ Nuts (Atwater and Bryant<sup>1</sup>)

1	Refuse.	Water.	Fat (Ether Extract).	Crude Fiber.	Protein (N X614).	Ash.	Nitrogen- free Extract.	Calories Per Pound.
Vegetables:								
Potatoes	20.0	62.6	0.1	0.3	1.8	0.8	14.4	310
Sweet Potatoes	20.0	55.2	0.6	1.0	1.4	0.9	20.9	460
Onions	10.0	78.9	0.3	0.7	1.4	0.5	8.2	205
Turnips	30.0	62.7	O. I	0.9	0.9	0.6	4.8	125
Cabbage	15.0	77 7	0.2	0.9	1.4	0.9	3.9	125
Lettuce	15.0	80.5	0.2	0.6	1.6	0.8	1.9	75
Corn, green	61.0	29.4	0.4	0.2	1.2	0.3	7.5	180
Peas, green	45.0	40.8	0.2	0.9	3.6	0.6	8.9	255.
Tomatoes		94.3	0.4	0.6	0.9	0.5	3.3	105
Asparagus		94.0	0.2	0.8	1.8	0.7	2.5	105
Beans, s OI.	7.0	83.0	0.3	1.8	2.1	0.7	5.1	180
Fruits:							_	
Apples°	25.0	63.3	0.3		0.3	0.3	9.9	220
Oranges	27.0	63.4	0.1	· .	0.6	0.4	8.52	170
Bananas	35.0	48.9	0.4	ċ '	0.8	0.6	13.6	300
Peaches	18.o	73 - 3	0.1	3	0.5	0.3	4.8	155
Raspberries	0.0	84.1	1.0	' '	1.7	0.6	12.62	310
Strawberries	5.0	85.9	0.6	<u></u>	0.9	0.6	5.7	175
Watermelons	59.4	37 · 5	0.1	oit	0.2	0.1	2.72	60
Grapes	25.0	58.0	I.2	ug T	1.0	0.4	11.3	335
Nuts:				•				
Almonds	45.0	2.7	30.2	I.I	11.5	1.1	8.4	1660
Brazil nuts	49.6	2.6	33 . 7		8.6	2.0	3 · 5 ²	1655
Chestnuts	24.0	4.5	5.3	2.0	8.1	1.7	54 - 4	1425
Cocoanuts	48.8	7.2	25.9		2.9	0.9	14.3 <sup>2</sup>	1413
Peanuts	24.5	6.9	29. I	1.9	19.5	1.5	16.6	1935
Pecans	46.3	1.5	37.9		5.1	1.0	8.22	1846
Walnuts	58.1	1.0	26.6	1.6	6.9	0.6	5.2	1375

<sup>&</sup>lt;sup>1</sup> The Chemical Composition of American Food Materials.

<sup>&</sup>lt;sup>2</sup> Includes crude fiber.

AVERAGE COMPOSITION OF SPICES (WINTON, OGDEN AND MITCHELL)

	ETHER EXTRACI.				Ash.	
Water.	Volatile.	Non- volatile.	Crude Fiber	Protein (N×64).	Total.	Sand (In- soluble in HCl)
Black pepper 11.96	1.14	8.421	13.06	12.052	4.76	0.47
White pepper 13.47	0.73	6.913	3.14	10.892	1.77	0.10
Cayenne pepper 5.73	1.35	20.15	22.35	13.67	5.43	0.15
Cinnamon 9.24	2.61	2.12	22.96	4.34	4.73	0.56
Ginger 10.44	1.97	4.10	3.91	7-74	5.27	0.44
Allspice 9.78	4.05	5.84	22.39	5.75	4.47	0.03
Cloves 7.81	19.18	6.49	8.10	6.18	5.92	0.06
Nutmegs 3.63	3.02	36.70	2.51	6.73	2.28	0.00
Mace	7.58	22.48	3.20	6.47	2.01	0.07

<sup>1</sup> Includes 6.72% piperine.

\*Drawing the Sample. It is of the utmost importance that the sample of food is carefully drawn and is so prepared for analysis that the portions weighed out for the individual determinations accurately represent the material. Failure to secure a proper sample renders the analysis worthless no matter how carefully the details of the method are followed.

It is particularly difficult to secure a representative sample when the quantity of the material is large, such as a ship load or several car loads. Such a quantity is seldom uniform throughout owing to various causes. For example a cargo of wheat may consist of different varieties grown by different farmers in different sections. Even the product of the same producer or manufacturer is likely to vary, at least in moisture content.

In order to secure an absolutely accurate sample of such a shipment it would be necessary first to thoroughly mix the whole product, which would be obviously impracticable. The usual procedure is to take portions of the material from dif-

<sup>&</sup>lt;sup>2</sup> Corrected for nitrogen as piperine.

<sup>3</sup> Includes 6.11% piperine.

ferent parts, mix these portions thoroughly, and remove suitable samples.

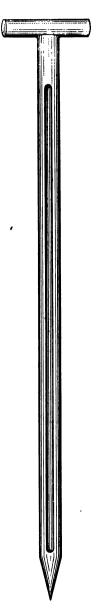
If the material is in bags a sampling tube (Fig. 22), may be used. This consists of a brass tube 2 to 3 ft. long in which is a slot extending from the conical tip nearly to the crosspiece serving as a handle. The tube is introduced into the bag with the slot on the under side, then turned so that the tube can fill, thus securing a core the entire length.

The sampling is best carried out in the presence of the interested parties, such as buyer or official inspector and seller, and the mixed sample divided so that each can have a portion for analysis with another portion in reserve to go to a disinterested chemist in case of dispute. If samples are sent from one party to another they should be under seal.

Samples of a pint or quart are usually sufficient, and fruit jars are well suited for containers.

When the product is in retail packages such a package may be assumed to be representative and true to label and may either be taken in its entirety for a sample or else mixed and sampled as described.

The sampling of succulent vegetables presents greater difficulties and need not be undertaken by the beginner. A quantity is weighed, sliced or chopped, and dried by artificial heat at a moderate temperature until brittle. Before cooling, the dried sample is ground, taking care to avoid mechanical loss, again weighed, and Fig. 22.—Sampling Tube.



bottled. In calculating the analysis of the original fresh material, of course account must be taken of the water lost in drying.

The student, after being provided with a sample of one of the foods named on p. 44 in a glass-stoppered bottle or fruit jar, should proceed as follows: Empty the sample onto a sheet of manilla paper and raise one corner after another, thus thoroughly mixing the material. By means of a steel spatula about

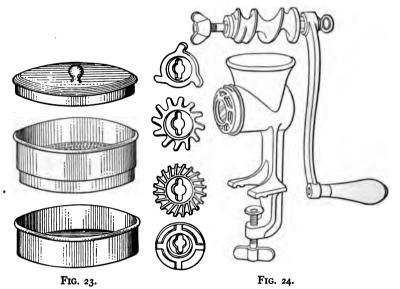


Fig. 23.—Sieve with Cover and Receiver. Fig. 24.—Universal Food Chopper.

r in. broad remove portions from different parts of the mixed substance to a 4-ounce wide-mouth glass-stoppered bottle until the latter is filled. This constitutes the subsample which is prepared for analysis; the remainder of the sample is held in reserve.

Another way is to divide the sample by halving and quartering, bottling each fraction separately.

\*Preparing the Subsample for Analysis. Flour, cocoa, most ground spices, and some other foods are in suitable condi-

tion for analysis without further preparation except thorough mixing; most foods, however, require grinding, so as to pass a sieve with a sheet metal bottom having round holes 1 mm. (equivalent to about  $\frac{1}{25}$  in.) in diameter or one with a bottom of No. 20 mesh wire cloth. The sieve should be provided with a cover and a receiver (Fig. 23).

The grinding may be done in an iron mortar, a coffee mill, or a "Universal" food chopper (Fig. 24), according to its nature, sifting from time to time until all passes through the sieve.

Care must be taken not to lose any of the material during grinding, as that would change the composition of the sample.

There are few materials that cannot be reduced to a powder with no apparatus other than a food chopper and an iron mortar. The iron mortar should be supported on a block which in turn rests on a firm support such as a cement floor or the portion of a floor over a girder of the building. To prevent loss of the brittle materials the top may be covered with a sheet metal disk with a hole in the middle large enough to admit the pestle (Fig. 25). Mills and other mechanical devices reduce the labor of grinding, but great care must

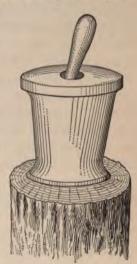


Fig. 25.—Iron Mortar with Guard.

be taken in cleaning off the adhering material to avoid loss. Exposure of the sample and consequent drying must be avoided.

When ground, mix the subsample thoroughly and bottle.

\*Care of Sample. Samples (and subsamples) should be kept in tightly stoppered bottles and analyzed as soon as possible after grinding. They should not be exposed to heat or to direct sunlight which cause the water to evaporate or condense in the neck of the bottle or on the inside of the stopper. Care should be taken not to jar the sample, which causes dry materials

to separate into strata of different densities. Before weighing out portions for analysis the contents of the bottle should be thoroughly mixed with a spoon (not a spatula), until they appear uniform. The portions should be weighed out as rapidly as possible to avoid loss or gain of moisture on the balance pan.

Conditions Affecting the Amount of Moisture in Foods. Fresh fruits and vegetables contain more moisture than all the other constituents taken together, and the same is true of most grains, seeds, leaves, roots, and other products before drying or curing. After drying by natural or artificial heat to a sufficient degree to insure keeping, the amount of moisture is reduced to about 15 per cent or less, depending on the process of drying and the humidity of the locality of storage. We speak of grain, flour, meal, bran, and other foods as being "air-dry" when they are in moisture equilibrium with the surrounding atmosphere. The percentage of moisture of an air-dry material is naturally higher when dried in a humid locality than in a dry one. If stored in tight containers such as tin boxes the moisture remains practically constant; if stored so as to allow access of air it changes somewhat from time to time, but the change is very gradual and cannot keep pace with daily fluctuations of the atmosphere.

Flour and meal, as usually milled and unless packed in tight containers, lose moisture during storage in most localities, while roasted coffee, biscuit, and various kiln-dried products gain.

Although a certain amount of moisture is unvoidable, an excessive amount is a detriment because (1) it causes spoilage, (2) it reduces the percentage of the dry matter or food proper, and (3) it unnecessarily increases the cost of transportation. Each per cent of excess water is tantamount to a per cent shortage in weight.

Consideration of Methods of Determining Moisture. Most of the methods for the estimation of moisture in foods depend on the loss in weight on heating. The temperature employed varies from 70° C. for saccharine substances containing invert

sugar to 110° for various foods. Commonly the temperature of boiling water is specified, the heating being carried out in a water oven (Fig. 6). The temperature in such an oven even near the sea level never reaches 100° C., being usually 97° to 99°, while in high altitudes such as Denver it is considerably lower.

As exposure to the air of the drying oven causes the oxidation of certain oils and other constituents, a gain in weight of such constituents offsets the loss in weight due to moisture. To obviate this error the drying should be performed *in vacuo* or in a current of dry hydrogen, the former being preferred for saccharine substances, the latter for natural foods and mill products.

The loss in weight on heating is not entirely water, as other volatile substances evident to the sense of smell are present in most foods, although the amount is usually too small to be separately determined. Most of the spices, however, contain notable quantities of volatile or essential oil which passes off with the water. Cloves contain 15 to 25 per cent of an essential oil, nutmegs and mace 3 to 10 per cent, and most of the other spices smaller quantities. In these it is the common practice to determine the total loss at 110° C. and correct the figures thus obtained for essential oil separately determined.

Heating is not always employed to remove the moisture. Benedict dries at room temperature in a sulphuric acid desiccator for some days. Grindley hastens the process by gently agitating the sulphuric acid during the drying, thus mixing the surface film—which soon becomes saturated with moisture—with the lower layers.

Again all methods do not depend on loss of weight after removal of moisture. The apparatus of Hoffman and of Brown and Duvel, used for the rapid determination of moisture in grain, are constructed so that the moisture driven off on heating with a petroleum oil in a flask is condensed and measured in a graduate.

The method selected for practice is that of drying at the temperature of boiling water in a current of dry hydrogen. It

not only, in the author's opinion, gives the most accurate results in the analysis of cereal and oil-seed products, tea, coffee, cocoa, and certain spices, such as cayenne pepper and mustard, containing small amounts of essential oil, but also involves details of manipulation particularly instructive for the student.

\*Method of Determining Moisture by Drying in Hydrogen. The apparatus (devised by the author 1) is shown in Fig. 26.

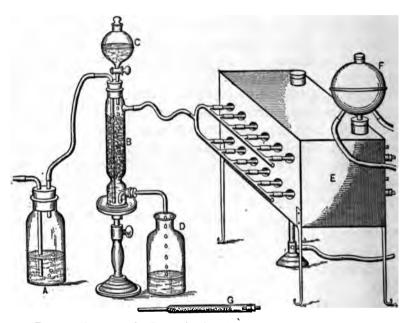


Fig. 26.—Apparatus for Determination of Water by Drying in Hydrogen.

The weighed portions of the materials are contained in glass tubes G which are heated in copper tubes soldered in the copper water oven E.

The oven is 22 cm. long, 18 cm. wide, and 18 cm. high, exclusive of the legs, which are 18 cm. long. The copper tubes are 17 mm. inside diameter. The glass drying tubes are not over 14 mm. outside diameter, fused but not flared at the end, and

<sup>&</sup>lt;sup>1</sup> Conn. Agri. Expt. Sta. Rep. 1889, p. 187.

should enter any of the copper tubes of the bath without binding. The length to the constriction is 15 cm., the total length 20 cm. Each end of the tube is provided with a cork. A small circle ground or etched with "diamond ink" on each tube serves for a lead-pencil number or other identification mark in place of gummed labels, which change in weight on heating.

A stream of hydrogen, purified by passing through nearly saturated sodium hydroxide solution in A and dried by c. p. concentrated sulphuric acid in B, is divided into twelve streams by means of a U-shaped metal tube with twelve offsets, one of the streams passing through each of the drying tubes. In order that the hydrogen may be evenly distributed, the mouth of each drying tube is fitted with a perforated cork through which passes a capillary exit tube of 0.5 mm. bore. The sulphuric acid used for drying the hydrogen falls drop by drop from the bulb C over the heads in B into the bottom of the jar, from which it automatically siphons out into D. The hydrogen passes to the bottom of the jar through a glass tube, bubbles through the acid, and rises through the beads, moist with fresh acid. The very thorough dehydration of the acid thus effected, doubtless, contributes to the accuracy of the results, which in flour and meal are about 1 per cent less than are obtained by drying in a dish in the cell of an ordinary water oven.

Six students should make duplicate determinations in the apparatus at the same time. On the day when the samples are ground there will be sufficient time to set up the apparatus, weigh out the portions into the tubes, and carry out the other preliminary details so that the next day can be devoted entirely to the drying process.

The following instructions should be strictly followed to insure success: Place in the funnel-shaped portion of the drying tubes a small wisp of cotton weighing but a few milligrams. Although cotton contains hygroscopic moisture, the amount present in such a small quantity will not appreciably affect the results. Mix the sample thoroughly in the bottle with an aluminum spoon and weigh out 2 gram portions on a balanced watch-

glass. If the watch-glasses do not weigh exactly the same, place the heavier on the left-hand pan, the lighter on the right-hand pan, and balance exactly with the rider. Introduce the weighed portions into the drying tubes through a small short-stemmed copper funnel (Fig. 27), using a camel's-hair brush to



Fig. 27.—Copper Funnel for Filling Drying Tubes, Nitrogen Flasks, etc.

remove the last particle from the watchglass and funnel. Weigh the tubes after introducing the substance (but not before), without the corks, then cork and hold until the next day.

Fill a large Kipp generator with granulated zinc and make up a supply of 20 per cent sulphuric acid ready to be introduced into the generator the next day. The sulphuric acid used in the drying jar

can be diluted for the generator. Place in A a sufficient quantity of nearly saturated sodium hydroxide solution to cover the lower end of the inlet tube, fill C with c. p. concentrated sulphuric acid, and see that the bath E contains enough water to cover the upper tier of tubes. Connect up the apparatus as shown in the cut.

On the following day, while the bath is heating to boiling, add the acid to the hydrogen generator and run for a time to expel all air, then pass the gas through A and B into the metal U-tube. Uncork the glass drying tubes, insert the corks with the capillary outlet tubes, place one after another in the tubes of the drying oven, connecting each at the same time with one of the offsets of the U-tube. When all are connected draw the drying tubes to the left until each outlet tube is well within the bath, thus preventing the clogging of the capillary openings with condensed moisture, and adjust the hydrogen current so that there is a steady and moderately rapid evolution. Adjust the stopcock of C so that the acid falls, one drop in about five seconds, over the beads.

After about one hour push the drying tubes toward the right so that the substance is well heated and the outer tubes

project 2 to 3 cm. Test each by lighting with a match. If the capillary openings are clear the hydrogen will ignite, usually with a distinct pop, and when the water has been largely expelled will burn with a miniature flame.

Four hours after starting the drying is complete. If, however, time is pressing, the tubes can be taken off a half hour earlier without appreciably affecting the results.

Cork each tube immediately after removing from the bath, cool fifteen minutes, place the weights on the pan corresponding to about 10 per cent loss (0.20 gram), remove the cork and finish the weighing as rapidly as possible, as the dry substance is very hygroscopic. Immediately after weighing cork tightly, as the dry material is to be used for extracting with ether. Calculate the loss in weight as the percentage of moisture.

Determination of Moisture in Spices. The preceding method cannot be used for spices owing to the volatile oil slowly driven off at 100°, which clogs the exit tubes. Such products are best weighed out into covered aluminum or tin dishes and dried to constant weight at 110° C. in a special oven. The loss sustained is due partly and, in the case of cloves, largely, to volatile oils which must be determined as described on p. 59 and the amount deducted.

Constituents of the Crude Fat or Ether Extract. The material extracted from dried natural vegetable substances by anhydrous ethyl ether is commonly known as fat or crude fat. A more exact term is ether extract, as substances other than fats and oils may be present. The composition of the vegetable fats and oils of commerce corresponds quite closely with that of the ether extract of the seeds and fruits from which they are derived, consisting largely of glycerides of the fatty acids with small amounts of phytosterol, coloring matter, and other minor constituents. The ether extract of tea, various pot herbs, hay, green vegetables, and other green parts of plants contains the blue and yellow coloring substances of the chlorophyl grains which impart to the extract a deep green color. Most spices contain essential oils and related resins which are soluble in

ether, but only the non-volatile resins remain in the ether extract after continued heating. A large part of the ether extract of black pepper consists of a crystalline nitrogenous substance, piperine.

Notwithstanding the variety of substances which may be present, the ether extract of most staple animal and vegetable foods is essentially true fat, i.e., glycerides of fatty acids, and the common use of the term fat as a synonym for ether extract causes little confusion.

In the chapter on fats and oils (p. 139), the chemical constitution of the glycerides will be considered.

Principles Involved in the Determination of Crude Fat. Not only ethyl ether, but chloroform, petroleum ether (purified gasolene), and other volatile solvents for fats and oils have been used in analytical processes. As the results with the different solvents do not always agree, it seems best to cling to ethyl ether, which has been used in obtaining most of the results recorded in the literature. As in the determination of fat in milk, anhydrous alcohol-free ether is employed, and the extraction is made on the dry substance, otherwise water- and alcohol-soluble constituents, notably sugars, will be extracted. Theoretically the extract as well as the substance before extraction should be dried in hydrogen to prevent oxidation of the fat, but practically this is not necessary, even in the case of the highly oxidizable fat from linseed meal, provided the drying is carried on only long enough thoroughly to remove the ether.

\*Method of Determining the Crude Fat. The Johnson extractor, which was used for the determination of fat in milk (p. 21), is best suited for extracting the crude fat from vegetable material. In place of the perforated cylinders, however, there should be provided inner tubes 135 mm. long and 22 mm. in (outside) diameter, with a constriction at the bottom for tying on a piece of filter paper backed by cheesecloth (Fig. 28). Cover the lower end of the inner tubes with one thickness each of filter paper and cheesecloth and fasten securely with strong linen thread wound tightly several times around in the constric-

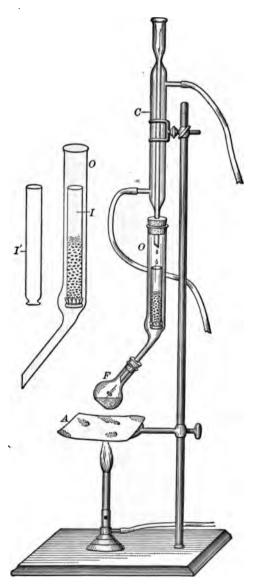


Fig. 28.—Johnson Fat Extractor.

tion and tied in a hard knot. To prevent the thread slipping while tying, first put one end twice around the other and draw up taut. The single knot thus formed will remain in place while completing the hard knot. Trim off the surplus paper and cloth with sharp-pointed scissors, preferably with curved ends.

Process. Place identification marks on the extraction flasks, weigh, and connect with the outer tubes of the extractors. Remove the plugs of cotton from the tubes containing the dried material remaining after the determination of moisture and

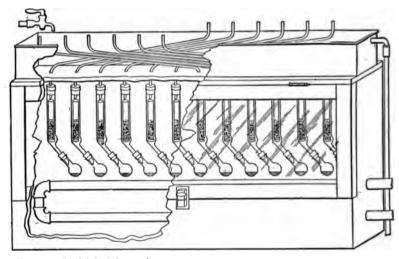


Fig. 29.—Multiple Johnson Fat Extractor with Heating Closet and Condenser.

transfer the material to the inner extraction tubes. If any appreciable amount adheres to the inner walls of the drying tube, brush off with a camel's-hair brush on the end of a glass rod. Shake off also any particles attached to the inner cotton plugs. Pour 8 to 10 cc. of anhydrous alcohol-free ether onto the material and connect the extractor with the condenser. Run the extraction for three and one-half hours. Turn off the heat, remove the inner tubes, and in their place introduce short test-tubes. Reserve the extracted residues for crude fiber determination.

On the following day turn on the heat and continue the heating until nearly all the ether has been driven off from the fat and condensed into the test-tubes. Dry the fat in a boiling water oven for one hour, cool for fifteen minutes, and weigh. Calculate the percentage of crude fat.

Fig. 29 shows a multiple apparatus for carrying on twelve determinations at the same time, devised by S. W. Johnson and modified by the author. The heating is performed by steam pipes, the glass door preventing loss of heat. The condensing tubes are of block tin cooled in a copper tank.

Modified Method Applicable to Spices. Extract 2 grams of the material, without drying, into a flask, which need not be weighed. After extraction transfer the ether solution to a weighed tinned lead, aluminum, or porcelain dish (p. 15), rinsing with ether, and allow to evaporate at room temperature, avoiding draughts which cause condensation of water in the dishes. Dry overnight in a desiccator and weigh, thus obtaining the joint weight of volatile and non-volatile ether extract. Heat first at 100° C., then at 110° C. to constant weight. The loss represents the volatile extract.

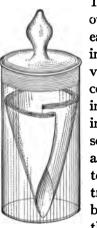
Nature of Crude Fiber. Vegetable tissues consist of cell walls and cell contents. The estimation of crude fiber gives us an approximate idea of the amount of organic cell-wall material which, in active cells, consists largely of cellulose and in older tissues of cellulose infiltrated with lignin (wood substance), suberin (cork substance), or other related substances. Silica, which is found in considerable amount in the straw and grain hulls of the cereals, although distinctly a cell-wall constituent, is not included in the crude fiber.

Defined as to the process employed in its determination, crude fiber is the substance or substances, other than mineral matter, insoluble in ether, boiling dilute sulphuric acid, and boiling dilute sodium hydroxide.

While cellulose is the chief material of the crude fiber, other substances are present in variable amount. On the other hand all the cellulose is not included, as it is somewhat soluble in the boiling acid used in the process of determination. Among the constituents of crude fiber other than those already named are small amounts of nitrogenous substances and pentosans.

While it is obvious that the term "crude" is significant and the crude fiber is a more or less indefinite mixture, still the determination is of very great value both in determining the feeding value of various materials as well as in detecting foreign substances. As "sand" is used to designate the mineral matter insoluble in acid, so "crude fiber" serves to describe the organic matter insoluble in certain reagents, and it is usually immaterial what silicates are present in the sand or what particular cell-wall substances occur in the crude fiber. Sand and crude fiber are the ingredients of foods of least value because of their insolubility.

# \*The Henneberg Method of Determining Crude Fiber.



Bottle with Filter Paper for Crude Fiber.

This process, 1 followed with few changes throughout the world, has come down to us from the early days of feeding experiments. It consists in boiling 2 grams of the ground material, previously extracted with ether, first with 1<sup>1</sup>/<sub>2</sub> per cent sulphuric acid, which converts the starch into soluble sugars and dissolves certain ash ingredients, and secondly with 17 per cent sodium hydroxide, which dissolves such proteins as resisted the action of the acid. If the material is not first extracted with ether, which treatment is impracticable in certain cases, the boiling with alkali saponifies most of the fat, the remainder being dissolved by a final wash-Fig. 30.—Weighing ing with ether. The crude fiber contaminated with mineral matter is dried and weighed. It is then burned and the weight of the residue of mineral matter deducted.

*Process.* Place in each of two cylindrical weighing bottles. 75 mm. high and 40 mm. in diameter, without a constricted <sup>1</sup> Land. Vers., 6, p. 497.

neck, a rolled-up 11-cm. filter paper folded ready for use and bearing a lead-pencil mark corresponding to that on the bottle and its stopper (Fig. 30). A lead pencil can be used on the ground surface of both the bottle and the stopper, thus avoiding permanent marks or numbers. Remove the stopper and place in a boiling water oven to dry.

Measure 200 cc. of water from a graduate into each of two 500-cc. Erlenmeyer flasks and mark the level with a gummed label. Remove from the extractors the inner tubes, containing the residues from the determination of ether extract, and allow the ether to evaporate. Empty the dry residues into the flasks and brush out any that may adhere with a camel's-hair brush on the end of a glass rod. All this should be done on the day when the extraction is performed as the crude fiber process, owing to the slow filtration after boiling with acid, is liable to require all the time of one laboratory period.

Heat a little more than 400 cc. of 114 per cent sulphuric acid solution and when the boiling-point is reached immediately pour into the two Erlenmeyer flasks up to the 200-cc. mark. Without delay heat over a gauze with a moderate-size flame, taking care to watch the flasks constantly and lower the flame to the smallest possible size at the first indications of boiling. If the liquid is allowed to boil vigorously it is almost sure to froth over and without warning thus ruining the determination. A glass tube bent at right angles at the end (Fig. 31), devised by Farrington, should be at hand so that a blast of air from the mouth can be instantly directed into the flask to prevent frothing. In order to reduce the flame to a small size without snapping down, the top of the Bunsen burners should be covered with caps of wire gauze (Fig. 32). It is well to keep gauze caps on all the burners used in food work, as low flames are often employed.

Keep the flames adjusted so that the liquid boils very gently and continue the boiling exactly thirty minutes. If these precautions are observed, not only will there be little danger of frothing over, but the substance will not crawl up far on the sides of the flask where it would be washed by condensed water and not subjected to the action of the acid. Any small amount that may crawl up may be brought down by very gentle rotation of the flask. Concentration of the acid will also be avoided and quite as effectually as by the use of a cumbersome reflux condenser recommended by some chemists.

At the end of the thirty minutes filter without delay on 11-cm. papers, selecting a quality known to filter rapidly. Keep the

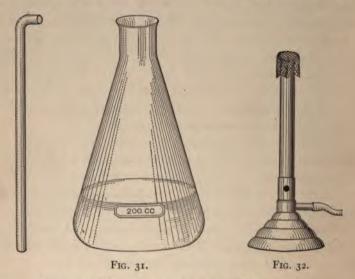


Fig. 31.—Flask and Tube for Crude Fiber Determination. Fig. 32.—Bunsen Burner with Wire Gauze Cap.

papers well filled with liquid. If they clog to such an extent that the filtration cannot be finished in say an hour, use a second paper or even a third. Rinse the flask once only, using a few cc. of hot water, but do not attempt to remove all the substance to the paper. The water used to rinse the flask is sufficient to wash the paper.

When the filtration is nearly finished heat to boiling in a neaker a little over 400 cc. of  $1\frac{1}{4}$  per cent sodium hydroxide solution. Spread out the paper (or papers), on a 12.5-cm. fun-

nel and rinse with the hot alkali back again into the flask used for the acid boiling. The alkali speedily removes the substance from the paper, leaving sufficient to rinse the funnel and wash down the sides of the flask. When the level of the mark on the flask is reached, heat to boiling and boil gently for thirty minutes exactly as in the acid boiling. As frothing during the alkali boiling is likely to take place with scarcely any warning, it is necessary carefully to control the heat and keep constant watch.

After fifteen minutes' boiling remove the weighing bottles with filters from the water oven, stopper, and at the end of the boiling, after they have cooled fifteen minutes, weigh them.

Filter on the weighed papers, rinse all the materials out of the flasks, and wash thoroughly on the papers, using hot distilled water. The alkali filtration of most substances proceeds rapidly. After the washing with hot water has removed the alkali as tested with litmus paper, wash twice with 95 per cent alcohol and three times with ether, taking care to direct the stream into the fiber, otherwise it may not penetrate the mass. Remove the funnels from the flasks and keep overnight in a warm room to facilitate evaporation of the ether.

On the next day, if there is no evidence of moisture in the fiber, carefully transfer the papers with fiber from the funnels to the weighing bottles, dry in the boiling water oven for three hours, stopper, cool fifteen minutes, and weigh.

Wrap each filter paper closely about the fiber and burn to whiteness at a bright red heat in a porcelain capsule or crucible. When cool the ash may be readily brushed off from the crucible and weighed on a balanced watch-glass. Correct the weight of this ash for any ash in the paper and deduct the weight thus corrected from the weight of the crude fiber. Calculate the percentage of crude fiber.

Nature of the Proteins. The proteins, formerly known as the albuminoids and later as the proteids, are alike important in vegetable and animal physiology. In addition to carbon, hydrogen, and oxygen, which are the only elements contained in the carbohydrates and fats, and which are built up by the plant from carbon dioxide taken in through the leaves from the air and water absorbed by the roots from the soil, all the proteins contain nitrogen, the most valuable constituent of soils and fertilizers. Most of them contain sulphur and some of them phosphorus. The earlier vegetable physiologists believed that only nitrogen combined as nitrates formed usually by the "nitrification" of other nitrogenous compounds was available for plants. The classical researches of Hellriegel showed, however, that atmospheric nitrogen was readily utilized by leguminous plants (peas, beans, clovers, etc.), through the agency of bacteria residing in the root nodules of these plants. The proteins derived directly or indirectly from plants serve in the animal body not merely as fuel for keeping the body warm and carrying on various muscular activities—the chief role played by the carbohydrates and fats-but also to make nerve and muscle. While the carbon is eliminated as carbon dioxide through the lungs, the nitrogen is excreted by the kidneys as urea.

Although the earlier chemists determined the percentages of nitrogen, carbon, hydrogen, oxygen, and sulphur in the proteins and learned that their molecular weights were very high, the real constitution of these substances remained a mystery until Fischer showed that they are complex compounds of amino acids, the so-called "building stones," which can be split off by special processes. This work and that of Osborne, in the United States, who has obtained crystals of certain proteins, have placed this complex group on a definite scientific basis.

While the quantitative separation of the individual proteins is rarely possible, the fact that all of them contain approximately 16 per cent of nitrogen enables the analyst to calculate the percentage of protein from the nitrogen content using the factor 6.25. The results thus obtained should be properly designated "crude protein," partly because the nitrogen content of the different proteins varies considerably and partly because other nitrogenous compounds such as amides (i.e., asparagin), and alkaloids (i.e., caffeine and piperine), are often present.

\*Determination of Crude Protein by the Kjeldahl Method. Apparatus. (1) Digestion Stand. The multiple apparatus shown in Fig. 33 consists of a cast-iron stand, a horizontal lead pipe for carrying off the fumes, and a battery of Bunsen burners.

If special apparatus is not available, the digestions can be made on ordinary lamp-stand rings over individual burners. The flask may be supported on a pipestem triangle, resting on

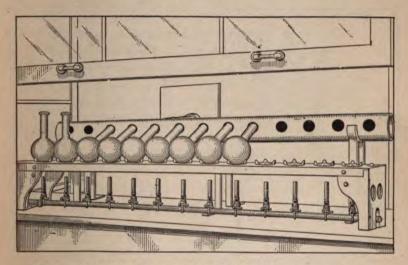


Fig. 33.-Multiple Digestion Stand for Kjeldahl Nitrogen Determination.

the ring, and the neck inclined at the proper angle against a clamp swung around to one side. The operation should be carried out in a hood with a good draught or Sy's suction device for removing the fumes from each flask employed.

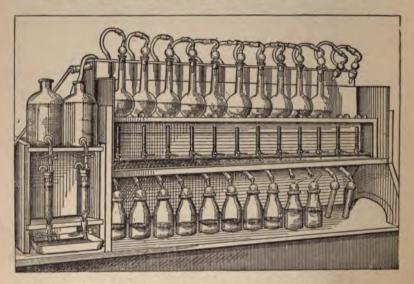
(2) Distilling Apparatus. Fig. 34 shows the Johnson multiple distilling stand with copper tank condenser and block tin tubes. At the left are shown bottles with swinging tubes for measuring the potassium sulphide and sodium hydroxide solutions which are not essential in the student laboratory.

# 66 NATURAL VEGETABLE FOODS AND MILL PRODUCTS

Lacking the special outfit the apparatus shown in Fig. 21, after removing the carbon dioxide delivery tube from the flask and closing the hole with a piece of glass rod, will answer the purpose.

(3) Burettes. Two 50-cc. burettes, one with a ball cock for the standard alkali, the other with a glass stopcock for the standard acid, are shown in Fig. 35.

The ball cock consists of a piece of black or red rubber



Frc. 34.—Multiple Johnson Distilling Apparatus for Kjeldahl Nitrogen Determination.

tubing closed by a glass bead somewhat larger than the inner diameter of the tubing. When pressed between the thumb and first finger passages are formed on opposite sides of the ball, allowing the liquid to run out. This form of cock is inexpensive, permits an accurate control of the flow, and avoids the cementing action of alkali on glass stopcocks.

The glass stopcock needs no explanation. To avoid the danger of the ground parts of burettes, separatory funnels, etc., becoming interchanged, lost, or broken, each may be

attached to its apparatus, as proposed by the author, by means of a small brass chain (Figs. 35, 93, and 104).

Fig. 36 shows the Squibb form of burette attached to a glass bottle containing the standard solution.

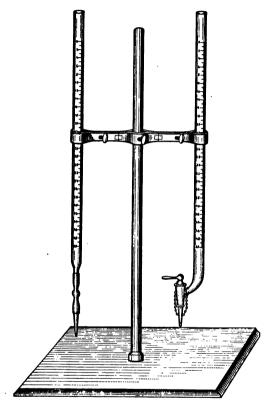


Fig. 35.—Burettes with Ball and Glass Stopcocks.

Reagents. (1) Concentrated Sulphuric Acid free from nitrogen.

- (2) Standard Tenth-normal Hydrochloric Acid.
- (3) Standard Tenth-normal Sodium Hydroxide Solution.
- (4) Sodium Sulphide Solution, 40 grams per liter.
- (5) Sodium Hydroxide Solution, nearly saturated.

- (6) Red Oxide of Mercury.
- (7) Potassium Permanganate.

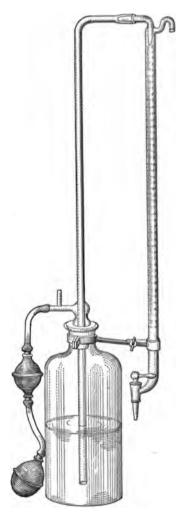


Fig. 36.—Squibb Burette with Filling Device.

- (8) Metallic Zinc, granulated, 20 mesh.
- (9) Cochineal Tincture. Digest 30 grams of the powdered bugs for some days in the cold with a mixture of 250 cc. of 95 per cent alcohol and 750 cc. of water, and filter.

Process. Weigh out 1 gram of the substance on a balanced watch-glass and transfer to a 600-cc. flat-bottom flask of the Iena type, add about 0.7 gram of red oxide of mercury (which can be measured from a small copper cartridge shell cut off to the proper length and provided with a wire handle), and 20 cc. of concentrated sulphuric acid free from nitrogen. Digest on the special stand shown in Fig. 33 at first with a low flame. The suffocating fumes of sulphurous acid, which are given off in large quantities during the first part of the heating, are carried off by the lead pipe into which the necks of the flasks enter through holes. After the densest of the fumes have been given off (fifteen to twenty minutes), raise the heat to the boiling-point but

avoid a flame high enough to impinge against the flask above the liquid. Continue the digestion until the liquid becomes light yellow, which requires usually two to three hours, turn off the flame and add from a small spoon, with gentle shaking, small quantities of potassium permanganate until a permanent brown or green color is acquired by the liquid. Great care should be exercised in handling the flask, as the boiling-point of the acid is about twice that of water. The nitrogen of the material will be completely converted into ammonium sulphate.

The remainder of the process can be finished on the next day. While the digestion is going on determinations of ash can be started.

On the next day add to a pint milk bottle from a burette an exactly measured quantity of tenth-normal standard hydrochloric acid, sufficient to slightly more than neutralize the ammonia formed by the digestion.

To find the suitable number of cc., divide the average per cent of protein, as given in the table on page 44, by 0.7. This allows for samples containing more than the average amount of protein and a reasonable excess.

Add a few drops of cochineal tincture or some other suitable indicator, such as a 0.02 per cent solution of methyl orange. Phenolphthalein cannot be used. Dilute to about 60 cc. and connect with one of the delivery tubes of the distilling apparatus (Fig. 34), adding water to the receiver if the tube does not dip below the surface.

To the liquid in the digestion flask add about 250 cc. of water, 25 cc. of 4 per cent sodium sulphide solution (to precipitate the mercury from any mercuro-ammonia compounds), and shake with a rotatory motion. Continue the shaking and add gradually nearly saturated sodium hydroxide to alkaline reaction, using litmus paper as indicator. Usually 30 to 40 cc. is sufficient. To avoid bumping add a pinch of coarsely powdered zinc with particles about the size of those of granulated sugar. Without delay attach to the distilling apparatus and light the flame beneath it. When the boiling-point is reached watch carefully so that the flame can be instantly turned out if there is danger of frothing over. Distill until about 250 cc. of

liquid have passed over, turn off the flame, and disconnect both the flask and the receiver tube. Rinse the latter and titrate the excess of acid with tenth-normal alkali. Deduct the volume of the alkali required in the titration from the volume of standard acid previously used and calculate the percentage of nitrogen by the factor 0.1401. Multiply the percentage of nitrogen by 6.25 to obtain the crude protein. In the case of wheat flour the factor 5.70 gives more nearly the true amount of protein.

Standard Acid and Alkali. Although no time is allowed in this course for preparing and standardizing solutions, the following details are given for the information of the student.

Hydrochloric acid of half-normal strength, that is containing as many grams of HCl per liter as half the molecular weight, is first prepared and standardized. This is used directly in finding the saponification number of fats and oils (p. 155) and, after dilution to tenth-normal strength, in the Kjeldahl process and in standardizing tenth-normal sodium hydroxide solution. The latter is required not only in the Kjeldahl process, but also in the determination of the Reichert-Meissl number (p. 157).

Standard Hydrochloric Acid. The concentrated c. p. acid has a specific gravity of 1.20 and contains about 39 per cent of the gas; one liter, accordingly, weighs 1200 grams and contains about 468 grams of HCl. One liter of half-normal acid contains (1.008+35.46) ÷ 2=18.234 grams of HCl. Calculated from these data, 39 cc. of the concentrated acid diluted to 1 liter will be approximately half-normal, but it is well to use 2 or 3 cc. more than the calculated amount, as it is more accurate to reduce with water to the exact strength, if too strong, than to add acid, if too weak. After the approximately half-normal acid has been prepared and mixed, determine its strength as follows:

Rinse a burette with a few cc. of the acid and fill to the zero mark. Draw off into a beaker with the greatest possible accuracy 20 cc. of the acid, dilute to about 150 cc., and

add silver nitrate solution drop by drop with stirring until a cloudy precipitate of silver chloride no longer forms. Heat nearly to boiling, continuing the stirring. After this treatment the silver chloride should be in a flocculent form beneath a practically clear liquid. Add a drop of silver nitrate to be certain that the reagent is in excess.

Decant the clear liquid onto a weighed Gooch crucible prepared as described on page 27. To the silver chloride remaining in the beaker add 100 cc. of boiling hot water with stirring, then decant onto the crucible as before. Repeat the addition of water and decantation twice, transfer the precipitate by means of the wash bottle jet to the crucible, clean off the last traces from the beaker with a "policeman," and wash with five portions of boiling water. Dry the crucible cautiously on a piece of asbestos paper, finally raising the heat until the silver chloride contracts to small volume and begins to melt on the edges. Cool in a desiccator and weigh.

The weight of HCl equivalent to the weight of AgCl is obtained by multiplying by 0.2544. This factor is the molecular weight of hydrochloric acid (36.468) divided by the molecular weight of silver chloride (143.34). The product multiplied by 50 gives the weight of HCl per liter (w). The number of cc. of water (V) necessary to add to a given volume (v) of the acid to reduce it to exactly half-normal strength is found by the formula:

$$V = \frac{w}{18.234} v - v$$

To prepare tenth-normal acid pipette into a graduated liter flask 200 cc. of the half-normal acid, make up to the mark with water and shake.

Standard Sodium Hydroxide Solution. Only a tenth-normal solution, that is, one containing 4.0008 grams of NaOH per liter, need be prepared. Weigh out quickly somewhat more than 4 grams (say 4.5 grams) of dry c.p. sodium hydroxide, prepared from the metal, dissolve in 500 cc. of water, and add

barium hydroxide solution sufficient to precipitate any carbonic acid present. Filter quickly through a plaited filter into a rooo-cc. graduated flask. Without washing the filter, make up to the mark and shake. Measure out 20 cc. of this solution from a burette into a beaker and titrate with tenth-normal hydrochloric acid, using a few drops of cochineal tincture as an indicator. By proportion calculate the volume of water necessary to add to a given volume of the solution to make it exactly half-normal, that is, so a given number of cc. neutralizes the same number of cc. of the tenth-normal acid.

Gunning-Arnold Modification of the Kjeldahl Method Applicable to Black and White Pepper. This method is used because the ordinary method does not give the full amount of nitrogen present in piperine. To 1 gram of the material add 1 gram of crystallized copper sulphate, 1 gram of red oxide of mercury, 15 to 18 grams of potassium sulphate, and 25 cc. of concentrated nitrogen-free sulphuric acid. Digest at a gentle heat with shaking until frothing ceases, then boil for three to four hours. In other respects proceed as in the regular Kjeldahl method, using, however, 50 instead of 25 cc. of sodium sulphide solution.

Piperine may be calculated from the nitrogen obtained by the Gunning-Arnold method in the ether extract from 10 grams of the pepper, using the factor 20.36.

The total nitrogen less that in the ether extract multiplied by 6.25 gives the corrected protein.

Constituents of the Ash of Vegetable Foods. Mayer, as an aid to the memory, gives the substances essential for plant growth as water, four acids (nitric, carbonic, sulphuric, and phosphoric), and four bases (potassium and iron oxides, lime, and magnesia). Of these, all but water, nitric acid, and carbonic acid are distinctly inorganic or ash constituents. In many fruits and vegetables the bases are partly combined with organic acid which burn to carbonates; in cereals and most seeds, however, there is sufficient sulphur and phosphorus to form sulphates and phosphates with the bases.

ASH 73

Although not essential for growth, chlorine and silica occur in many vegetable products and aluminum and some other inorganic elements are often present in small amount.

The ash also contains sand and other extraneous dirt which become attached to the plant during growth or handling. So-called pure ash is ash corrected for sand, carbon dioxide, and unburned carbon or charcoal.

\*Determination of Ash. Weigh 2 grams of the material on a watch-glass and place in a weighed porcelain crucible, wide form, 40 mm. in diameter. Bulky material may be added in several portions during the incineration.

If a small platinum dish or crucible is at hand it will be

found by far the best for ash determination, but the expense is prohibitive if it is to be used only for this course.

Heat gradually on a piece of asbestos paper to dull redness and continue to heat at that temperature until further heating does not appear to reduce the bulk of the residue or change its appearance. The ashing is best finished in a muffle furnace, preferably of the electric type (Fig. 37), but

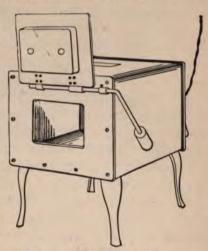


Fig. 37.—Hoskins Electric Furnace.

lacking this, may be carried out on the asbestos paper, using a second piece to cover the crucible and thus raise the heat to uniform dull redness. If difficulty in securing a white or light gray ash is experienced, remove from the furnace, cool, add I or 2 cc. of water, evaporate to dryness, and again ignite. By this treatment particles of carbon will be liberated from any salts which may have become fused about them and thus rendered more readily combustible. Overheating retards rather than

hastens the burning, furthermore at a bright red heat alkali chlorides are volatilized and carbon dioxide is expelled from calcium carbonate. Finally cool in a desiccator and weigh.

\*Nitrogen-free Extract. The constituents of vegetable substances, other than those of the five groups which we have learned to determine, are known collectively as the nitrogen-free extract. The most important substances of the group are the carbohydrates, including starch, sugars, and gums. Other substances which, if present, belong in this group are organic acids, tannin substances, and various mimor constituents.

The percentage of nitrogen-free extract is obtained by difference, that is, the sum of the percentages of water, ether extract, crude fiber, crude protein, and crude ash is subtracted from 100.

In the case of the cereals and some other starchy products, the results obtained represent fairly accurately the amount of starch and related carbohydrates present; in some other cases they have less definite significance.

Chemical Properties of Starch. The formula  $C_6H_{10}O_5$ , ascribed by the earlier chemists to starch, represents the ratio of the atoms of the three elements in the molecule rather than the actual molecular constitution. It is now considered that the molecular weight of starch, as well as of *Dextrin* and *Cellulose*, which also contain the three elements in the ratio of 6:10:5, is, like the molecular weight of the proteins, very large, the hypothetical formula of starch being given as  $(C_6H_{10}O_5)_{200}$  and of dextrin as  $(C_6H_{10}O_5)_{40}$ . However great may be the scientific importance of determining the exact molecular constitution, the simple formula  $C_6H_{10}O_5$ , which represents a molecular weight exactly  $\frac{9}{10}$  of that of Dextrose  $(C_6H_{12}O_6)$ , suffices for analytical purposes.

Starch is the most important carbohydrate occurring in the vegetable cell. It is the first visible product of photosynthesis whereby the carbon dioxide of the air and the water drawn up through the roots are combined in the leaf, by the action of sunlight and through the agency of the *Chlorophyl*. As fast as it is formed in the leaf it is redissolved and moved to other

parts of the plant. In many seeds, roots, tubers, and barks it is deposited as reserve material in the form of granules the microscopic characters of which are considered in Chapter V.

Starch is dissolved by the enzymes of malt (Diastase), saliva (Ptyalin) and pancreas (Pancreatin), with the formation of Maltose. Heated with dilute acids other carbohydrates (soluble starch, amylodextrin, erythrodextrin, achroodextrin, maltodextrin), are believed to be formed successively, the blue color with iodine changing gradually to red and the red color in turn becoming more and more faint and finally disappearing entirely. The final product of this acid conversion is dextrose, and on this principle are based the common analytical processes. The Pentosans and to some extent the Cellulose of the cell walls are also converted into dextrose on heating with dilute acid. hence if considerable amounts of these are present the starch must first be dissolved out by malt extract (diastase), saliva, or some other solvent which acts only on the starch. In the socalled diastase method malt extract is employed. The dextrose obtained by the acid conversion whether by direct treatment or after digestion with malt extract is best determined by copper reduction, employing a boiling alkaline solution of copper sulphate and Rochelle salts, known as Fehling solution. Dextrose, levulose, maltose, and lactose reduce in different degrees the copper of this solution to copper suboxide (Cu<sub>2</sub>O), whereas sucrose and the dextrins are practically non-reducing.

\*Determination of Starch in Wheat Flour. The diastase method, involving, as it does, not only the preliminary treatment with malt extract, but also the determination of the correction due to the soluble carbohydrates in the malt extract, is too difficult for the beginner. Fortunately ordinary wheat flour contains so little cellulose and pentosans that direct treatment with acid by Sachsse's method <sup>1</sup> introduces no appreciable error. Soluble carbohydrates are present only in small amount so that preliminary washing with water or dilute alcohol to remove these may be omitted. Proceed in duplicate as follows:

<sup>1</sup> Chem. Centralbl., 1877, p. 732.

Method. Weigh out 2.5 grams of wheat flour on a watch-glass, transfer to a flask of about 500-cc. capacity, add 200 cc. of water and 20 cc. of 25 per cent hydrochloric acid (sp.gr. 1.125), and rotate the flask until the flour is evenly distributed and there are no lumps on the bottom. Heat to boiling and boil very gently for two hours, replacing, from time to time, any water lost by evaporation, if perceptible. While the solutions are boiling pack two porcelain Gooch crucibles with an asbestos felt \frac{1}{4} in. thick, wash thoroughly with water to remove fine particles of asbestos, then with alcohol and with ether, dry thirty minutes in a boiling water oven, cool in a desiccator, and weigh.

The asbestos (amphibole), which must be of the grade specially furnished by dealers for copper reduction work, is prepared in advance for the use of the class. It is first digested with  $\mathbf{r}:3$  hydrochloric acid for two or three days and washed free of acid. It is then treated for a similar period with caustic soda solution and for a few hours with hot Fehling solution. After washing free of alkali it is digested with nitric acid for several hours and washed free of acid. A quantity of the pulp is shaken with water and the crucible loaded while it is in suspension.

On the day following the digestion of the flour with acid add a few drops of phenolphthalein solution and 20 per cent sodium hydroxide solution to slight alkaline reaction as shown by the pink color. Add hydrochloric acid, drop by drop, until the pink color just disappears and make up to 500 cc. in a graduated flask. Shake and filter through a dry paper into a dry flask.

Determine dextrose by the Munson and Walker method <sup>1</sup> as follows:

Pipette into a 400-cc. beaker 25 cc. of copper solution (34.639 grams c. p. crystals of copper sulphate dissolved in water and diluted to 500 cc.), 25 cc. of alkaline tartrate solution (173 grams of c. p. crystallized Rochelle salts and 50 grams of sodium hydroxide dissolved in water and diluted to 500 cc.), and 50 cc. of the filtered flour extract. Cover with a watch-glass, heat on an

<sup>&</sup>lt;sup>1</sup> Jour. Amer. Chem. Soc., 1906, 28, p. 163.

asbestos gauze over a Bunsen burner with the flame so regulated that boiling begins in four minutes, and boil for exactly two minutes. Filter at once on one of the weighed Gooch crucibles, using suction. Transfer the cuprous oxide to the crucible and wash thoroughly with water at 60° C., then wash with 10 cc. of alcohol, and finally with 10 cc. of ether. Dry thirty minutes in a boiling water oven, cool in a desiccator, and weigh. In Munson and Walker's table (pp. 213-221), find the amount of dextrose corresponding to the weight of copper suboxide obtained, multiply this weight by .9 to convert into the weight of starch, divide by .25 (the weight of flour corresponding to 50 cc. of the solution or  $\frac{1}{10}$  of the total amount weighed out), and multiply by 100, thus obtaining the percentage of starch.

Pentosans in Mill Products. These substances (xylan, araban, etc.), bear the same relation to the pentose sugars (xylose, arabinose, etc.) as starch does to dextrose. Thanks to the researches of Tollens, Kröber, and others, they may be determined by conversion into furfural by distillation with hydrochloric acid and precipitation of the furfural as phloroglucide by phloroglucinol.

Flour Testing and Analysis. Through the efforts of Snyder and others, the work of the flour laboratory has become of great practical importance. In addition to the constituents already considered in this chapter, determinations are made of Gluten (wet and dry), Acidity, Absorption (water-absorbing power), and other physical characters. Of special value are scientific Baking Tests, involving the volume, flavor, texture, and color of the loaf.

Bleaching of flour with nitrogen peroxide is detected by quantitative determinations of *Nitrites*, and *Gasoline Color Value* (the color extracted by gasoline) and bleaching with chlorine is detected by estimation of the *Chlorine in the Fat*, *Iodine Number of the Fat*, and the gasoline color value.

## YEAST AND BAKING POWDER

Function of Aerating Agents. Yeast and baking powder are useful merely to generate carbon dioxide. The gas in its efforts to escape converts the dough, which otherwise would remain a soggy mass, into a light and porous loaf. Like extracts, spices, tea, and coffee, they have practically no food value.

In yeast leavening the alcohol, which, together with the carbon dioxide gas, is formed from the sugars, is either driven off in baking or else remains behind in such small quantities as to be negligible; in leavening by baking powder, however, the by-products of the reaction are appreciable quantities of fixed salts with more or less marked physiological action.

Yeast whether dry or compressed consists of an organism, one of several varieties of the species Saccharomyces cerevisiæ, mixed usually with an inert material such as starch, or meal. The best variety for bread-making is top yeast (Fig. 98), obtained from distilleries. Top yeast is also used in ale brewing. Bottom yeast (Fig. 97), such as is used in lager beer brewing, is considered inferior for bread-making. The plant belongs to the unicellular fungæ and reproduces by budding.

The aerating value of yeast is tested by the amount of carbon dioxide evolved from a sugar solution to which a certain weight of the sample has been added. Living yeast cells are distinguished from the dead cells by mounting in a very dilute solution of fuchsin or some other coal-tar color and microscopic examination. The live cells are not stained, whereas the dead cells absorb the dye and are readily distinguished from the others by their color.

The chemical reactions involved in fermentation are considered on page 168.

Baking Powder consists of a dry mixture of sodium bicarbonate with tartaric acid, potassium bitartrate (cream of tartar), calcium acid phosphate, sodium aluminum sulphate (soda alum), or aluminum sulphate. Starch is usually present to prevent deterioration. The reactions involved and the fixed products remaining in the bread will be understood from the following equations:

(1) 
$$H_2C_4H_4O_6 + 2NaHCO_3 = Na_2C_4H_4O_6, 2H_2O + 2CO_2$$
Tartaric acid Sodium Sodium tartrate Carbon dioxide

(3) CaH<sub>4</sub>(PO<sub>4</sub>)<sub>2</sub>+2NaHCO<sub>3</sub>

Calcium acid
phosphate

Sodium
bicarbonate

(5) 
$$Al_2(SO_4)_3 + 6NaHCO_3 = Al_2(OH)_6 + 3Na_2SO_4 + 6CO_2$$

Aluminum Sodium Aluminum Sodium Carbon dioxide sulphate dioxide

There can be no doubt as to the value of baking powder in yielding a light, easily masticated loaf, but how far this is offset by the physiological action of the residual products of the reaction opinions differ. Of the residual products, sodium tartrate, potassium sodium tartrate (Rochelle salts), disodium hydrogen phosphate, and sodium sulphate (Glauber's salts) are cathartics well known in medicine. The quantities ordinarily eaten in baking powder bread or in cake are much less than the medicinal doses.

\*Material for Laboratory Practice. Three baking powders, one of the cream of tartar type (Royal, Cleveland's, Price's, etc.), one of the phosphate type (Horsford's), and one of the alum or alum-phosphate type (K. C., Calumet, etc.), should be provided for qualitative tests, which will require but a short time.

Quantitative determinations require no little time and skill, and need not be attempted. Of special importance are estimations of total and available carbon dioxide, that is, the amount of gas liberated by acid and water respectively. In

both cases the carbon dioxide, freed from water by first passing through an inverted condenser and then through a tube containing calcium chloride, is absorbed either in a "potash bulb" containing caustic alkali, or else in a U-tube containing a fused mixture of sodium hydroxide and calcium oxide. known as soda-lime.

\*Test for Sulphates. Boil about 1 gram of the powder in a beaker with 100 cc. of water and 3 cc. of concentrated hydrochloric acid until a nearly clear solution is obtained. portion of the liquid in a test-tube add a few drops of barium chloride solution. If a copious precipitate forms, the powder contains alum, aluminum sulphate or else calcium sulphate filler.

\*Test for Phosphates. To another portion of the solution obtained as described in the preceding paragraph, while still hot, add ammonium molybdate solution. The formation of a bright yellow precipitate of ammonio-phosphomolybdate shows the presence of acid phosphate. If neither sulphates nor phosphates are present the powder may be assumed to belong to the tartaric acid or cream of tartar class, the two being closely related.

\*Leach Test for Aluminum Salts. This test 1 depends on following reaction:

Burn about 2 grams of the powder in a platinum dish at a dull red heat. It is not necessary to secure a white ash, as a small amount of unburned carbon does not interfere with the test. Extract with boiling water and filter. Add to the filtrate sufficient ammonium chloride solution to produce a distinct odor of ammonia. A flocculent precipitate indicates aluminum.

If calcium phosphate is present in the ash it will be insoluble in water; sodium or potassium phosphate, if present, will go into

<sup>&</sup>lt;sup>1</sup> Mass. State Board of Health, 31st Ann. Rep., 1899, p. 638. Leach's "Food Inspection and Analysis," Third ed., p. 343.

solution, but the phosphoric acid will only precipitate when an aluminum salt is present.

If the student is not provided with a platinum dish, this test may be performed in his presence by the instructor or may be omitted.

\*Test for Starch. Add a solution of iodine in potassium iodide to the dry powder or the pasty liquid obtained by boiling with water. The characteristic blue color indicates starch. The variety of starch may be identified under the microscope.

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#### CHAPTER V

# MICROSCOPIC EXAMINATION OF VEGETABLE FOODS

#### Introduction

Province of Microscopic Examination. The methods for the determination of the six groups of constituents of ground vegetable substances, detailed in Chapter IV, although very important in measuring the food value of the substances and in forming an opinion as to their purity, are of no decisive value in identifying unknown substances either singly or in mixtures. By these methods the analysis of different cereal products and various mixtures often shows practically the same composition, and the same is true of a variety of oil-seed products and other vegetable substances. Determination of starch, although serving to distinguish starchy from non-starchy products and those with high starch content from those with a low percentage, throws no light on the source of the starch.

A study of the histological structure, commonly known as microscopic examination, on the other hand, furnishes decisive information as to the nature and source of the constituents of vegetable products, thus supplementing the chemical analysis. Each cereal, oil seed, spice, and fruit, as well as each weed seed and common vegetable adulterant, has definite microscopic characters which permit in almost every instance its identification in the finest powders.

While some experience in the use of the compound microscope and an elementary knowledge of vegetable histology are desirable, it has been the author's experience that a careful student in eight exercises can gain an insight into the subject sufficient to enable him to prosecute intelligently further studies.

Microscopical examination is also of great importance in the examination of drugs, textile fibers, paper, and other technical products and, as in the case of foods, should be carried on in conjunction with chemical analysis.

Histological work is distinctly botanical and should not be confused with chemical microscopy or microchemical analysis, which is a branch of qualitative analysis based on the reactions observed under the microscope. The student wishing to pursue this subject is referred to Chamot's Chemical Microscopy.

Construction of the Microscope. The optics and detailed

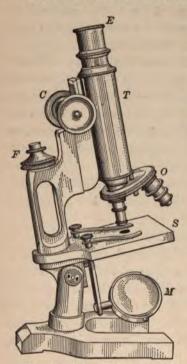


Fig. 38.—Microscope for Food Examination.

construction of the microscope, as of the polariscope, is a special subject for which the student or even the skilled microscopist has little need. Only a brief description of the instrument such as seems essential for practical work is here given.

Fig. 38 shows a simple form of compound microscope suited for the examination of food products. The stage S carries the slide or glass slip on which the material is mounted. The magnifying parts are (1) the objective O, of which two are shown in the figure mounted on a double nosepiece, a device for instantly throwing one objective into service and the other out, thus changing the magnification, and (2) the eyepiece or ocular E, consisting of lenses mounted

in a metal cylinder which is introduced in the top of the tube T of the microscope. C is the coarse and F the fine adjustment for focusing on the mount. The mirror M is mounted

below a hole in the stage on swinging supports so that it can be adjusted to cast a bright light on the material under examination, which otherwise, when magnified, would be too dark to show its structure. The light is tempered by different size holes or by a so-called iris diaphragm. For some kinds of work a substage condenser mounted above the mirror is essential. Eyepieces of different powers are also provided.

Microscopic Accessories. Micrometer. Each microscope should be provided with an eyepiece micrometer consisting of a

scale ruled on a glass disc mounted in a special eyepiece. This is used for measuring starch grains and other minute objects.

Polarizing Apparatus. In addition to the microscopes for the use of the individual students one equipped with polarizing apparatus should be provided for the class. In

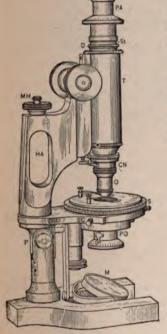






FIG. 40.

Fig. 39.—Chamot Polarizing Microscope. PO polarizer; PA analyzer. Fig. 40.—Dropping Bottle for Microscopic Reagents.

the polarizing microscope one of the Nicol prisms (the polarizer), is mounted below the stage and the other (the analyzer) in the microscope tube or above the eyepiece. One or the other of

these is in a revolving mount so that the axes of the two can be crossed as in the saccharimeter. Fig. 39 shows the Chamot polarizing microscope specially designed for microchemical analysis, but well suited for the examination of starch grains with polarized light. By adding a double nosepiece and a device for swinging the polarizer out of the axis, when ordinary illumination is desired, this same microscope can be used in food examination, thus avoiding the duplication of instruments for courses in chemical microscopy and food analysis.

Slides. Slips of glass,  $3 \times 1$  in., for mounting materials for observation.

Cover Glasses. Circles of very thin glass  $\frac{3}{4}$  in. in diameter.

Dropping Bottles (Fig. 40) for 5 per cent sodium hydroxide solution and iodine in potassium iodide solution (0.05 gram iodine, 0.2 gram potassium iodide, 15 cc. water).

\*Calibration of Micrometer. The student should calibrate his eyepiece micrometer partly to gain experience in finding an object under the microscope and in focusing and partly for a clearer understanding of the nature and use of the micrometer scale. The scale is usually graduated in millimeters and tenths of a millimeter, but this has no significance, as the material under examination and the scale are magnified to different degrees, and the magnification of the material differs with the objective. For the calibration a stage micrometer is used. This consists of a slide on which is etched an accurate scale with ultimate divisions o.o. mm. apart.

Set the microscope in front of a north window or at least where it will not be in direct sunlight. Place the stage micrometer on the microscope stage, and introduce the eyepiece micrometer in the tube of the instrument.

Draw out the microscope tube to standard length (usually 160 mm.). Using the low-power objective and eyepiece micrometer find the scale on the stage micrometer with the aid of the concentric circles which surround it, then, by moving the stage micrometer and turning the eyepiece micrometer, superimpose one scale on the other and count the number of ultimate divisions

below a hole in the stage on swinging supports so that it can be adjusted to cast a bright light on the material under examination, which otherwise, when magnified, would be too dark to show its structure. The light is tempered by different size holes or by a so-called iris diaphragm. For some kinds of work a substage condenser mounted above the mirror is essential. Eyepieces of different powers are also provided.

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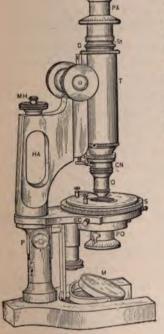






FIG. 40.

Fig. 39.—Chamot Polarizing Microscope. PO polarizer; PA analyzer.
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the polarizing microscope one of the Nicol prisms (the polarizer), is mounted below the stage and the other (the analyzer) in the microscope tube or above the eyepiece. One or the other of

the water mount thus prepared and, if desired, again after treatment with a reagent such as iodine solution to stain the starch grains or sodium hydroxide solution to dissolve the starch and proteins and clear the tissues.

Permanent mounts are not necessary for the work outlined in this chapter. The medium used for permanent mounts is usually Canada balsam, glycerine jelly, or glycerine, the cover glass in the latter case being fastened to the slide by a ring of cement.

Observation of the Mount. Adjust the mirror until the field is well illumined. Place the mount on the stage so that the material is over the middle of the hole. With the shorter or low-power objective in position, lower the tube carefully by the coarse adjustment until it nearly touches the cover glass, then, looking through the eyepiece, raise the tube until some part of the material or else a scratch on the glass comes into focus. Examine first with the low-, then with the high-power objective, taking care not to hit the cover glass. Move the slide with the left hand to bring different parts of the mount in the field and focus with the right hand. As a new focus must be found for each change in position of the slide and for parts of the mount at different depths in the same position, the fingers on the fine adjustment are kept continually busy. If the field is too light, use a smaller hole in the diaphragm; if too dark, use a larger one. Although theoretically more light should be used for the high- than the lower-power objective, after a few trials a degree of illumination can be found which will answer for both powers, and the attention can be confined to the details of moving the slide, focusing, and observation.

# MICROSCOPY OF STARCHES

Nature of Starch Grains. Starch occurs widely distributed in the vegetable kingdom as reserve material in the form of minute grains or granules. In seeds the starch is laid up as food for the plants' progeny—the young plant—to be used while it is reaching up through the soil and before it forms chlorophyl

below a hole in the stage on swinging supports so that it can be adjusted to cast a bright light on the material under examination, which otherwise, when magnified, would be too dark to show its structure. The light is tempered by different size holes or by a so-called iris diaphragm. For some kinds of work a substage condenser mounted above the mirror is essential. Eyepieces of different powers are also provided.

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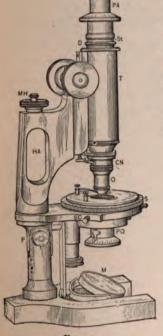






FIG. 40.

Fig. 40.—Chamot Polarizing Microscope. PO polarizer; PA analyzer.

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both cases the carbon dioxide, freed from water by first passing through an inverted condenser and then through a tube containing calcium chloride, is absorbed either in a "potash bulb" containing caustic alkali, or else in a U-tube containing a fused mixture of sodium hydroxide and calcium oxide, known as soda-lime.

\*Test for Sulphates. Boil about 1 gram of the powder in a beaker with 100 cc. of water and 3 cc. of concentrated hydrochloric acid until a nearly clear solution is obtained. To a portion of the liquid in a test-tube add a few drops of barium chloride solution. If a copious precipitate forms, the powder contains alum, aluminum sulphate or else calcium sulphate filler.

\*Test for Phosphates. To another portion of the solution obtained as described in the preceding paragraph, while still hot, add ammonium molybdate solution. The formation of a bright yellow precipitate of ammonio-phosphomolybdate shows the presence of acid phosphate. If neither sulphates nor phosphates are present the powder may be assumed to belong to the tartaric acid or cream of tartar class, the two being closely related.

\*Leach Test for Aluminum Salts. This test 1 depends on following reaction:

Burn about 2 grams of the powder in a platinum dish at a dull red heat. It is not necessary to secure a white ash, as a small amount of unburned carbon does not interfere with the test. Extract with boiling water and filter. Add to the filtrate sufficient ammonium chloride solution to produce a distinct odor of ammonia. A flocculent precipitate indicates aluminum.

If calcium phosphate is present in the ash it will be insoluble in water; sodium or potassium phosphate, if present, will go into

<sup>&</sup>lt;sup>1</sup> Mass. State Board of Health, 31st Ann. Rep., 1899, p. 638. Leach's "Food Inspection and Analysis," Third ed., p. 343.

solution, but the phosphoric acid will only precipitate when an aluminum salt is present.

If the student is not provided with a platinum dish, this test may be performed in his presence by the instructor or may be omitted.

\*Test for Starch. Add a solution of iodine in potassium iodide to the dry powder or the pasty liquid obtained by boiling with water. The characteristic blue color indicates starch. The variety of starch may be identified under the microscope.

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others (wheat, rye, barley). It is located in the geometric center of round and polygonal grains, in one end of ovoid grains. In legumes it is elongated.

Rings. In some grains rings, concentric about the hilum, are distinct, in others scarcely evident.

Aggregates. The grains in certain products are often united to form aggregates of a few (cassava) or numerous individuals (rice, oats, buckwheat).

Polarization Crosses are more or less evident with crossed Nicol prisms (Fig. 42). Except for the crosses the grains stand out brilliantly white, contrasting strongly with the dark field.



Fig. 48.—Cassava Starch. ×300. (Moeller.)

Because of these phenomena the grains are considered to be sphero-crystals.

\*Examination of Typical Starches. The following starches should be examined: Wheat, oat, bean, corn (maize), potato, and cassava (tapioca). Material suitable for the examination of the first three is obtained by cutting open wheat and oat kernels and ordinary white or navy beans, and scraping out a portion of

the powdery interior. Corn starch is sold in every grocery store. Potato and cassava starch are obtainable from dealers in chemical supplies, although uncooked potatoes and dried cassava roots answer the purpose quite as well.

A single laboratory period is sufficient for the study of these six forms of starch grains. Some of them will be seen again in the examination of various vegetable foods.

Mount in water and, using first the low- and then the highpower objective, note the shape of the grains, the character of the hilum (if visible), whether or not rings are evident, and the presence or absence of aggregates. Next, using the eyepiece micrometer, measure the diameter or length of the largest grain found. Finally, with the polarization microscope observe the distinctness of the crosses and their place of intersection.

To study the action of iodine on the grains place a drop of the reagent on one side of the mount and draw it under the cover glass by means of a piece of filter paper held on the opposite side so as to suck out a portion of the water.

The chief characters are shown in figures 43 to 48, inclusive, or are given in the following table:

	Wheat	Oat	Bean	Maize	Potato	Cassava
Form	Lenticular	Polygonal	Elliptical or Bean- shaped	Polygonal	Pear- shaped	Truncated
Maximum Size	50μ	10μ	60µ	35⊭	100μ	35µ
Hilum	Central, small	Central, small	Elongated, large	Central, large	Eccentric in small end of grain	Central, distinct
Rings	None or indistinct	None or in- distinct	Distinct	None or in- distinct	Distinct:	Indistinct
Aggre- gates	None	Numerous, up to 100 grains		None .	None	Present, mostly 2 to 3 grains
Polariza- tion Crosses	Indistinct	Distinct	Distinct	Distinct	Distinct	Distinct

# MICROSCOPY OF TYPICAL FOODS

\*Materials for Laboratory Practice. The following unground crude products should be provided: Wheat, rye, corn (maize), oats, buckwheat, peas, cotton seed, linseed (flax seed), black pepper, cayenne pepper, cinnamon (cassia) bark, ginger root, coffee beans, cocoa beans, and tea; also the following ground products for use in practice mixtures: Wheat flour, ground wheat

bran, ground rye bran, corn (maize) meal, ground oatmeal, buck-wheat flour, ground peas, cotton seed meal, linseed meal, ground black pepper, ground cayenne pepper, ground cinnamon, ground ginger, ground coffee, and cocoa. All of the ground products should be fine enough to pass at least a r mm.  $(\frac{1}{25}$  in.) sieve. Naturally the three kinds of flour and the cocoa will be impalpable powders.

Six laboratory periods should be given up to studying the general structure of the crude products. After this practice a day may be devoted to the identification of the ground products, both singly and in mixtures, submitted by the instructor.

\*Wheat. All of the true cereals are dry, one-seeded fruits





Fig. 49.—Wheat. Grain in Longitudinal Section and Entire. X8. (SCHUMANN.)

consisting largely of the starchy seed, the fruit coat or *Pericarp* being represented only by the outer bran layers. If a wheat kernel is examined with the naked eye it will be seen that on one side is a deep cleft extending the entire length of the kernel while on the other side at the base is a depression marking the location of the embryo or germ beneath. At the apex is a beard of fine hairs visible under a lens.

If a kernel is cut with a penknife into halves, through the cleft (Fig. 49), it will be seen from an examination of the cut surface with the naked eye, that the hard mass within the bran coats consists in large part of the *Endosperm* which, tested with a drop of iodine in potassium iodide solution, turns deep blue, showing that it is rich in starch. This starch is reserve

WHEAT 95

food for the plantlet while beneath the soil. White flour is made from the endosperm. The Embryo (e) does not contain starch, but is rich in oil and proteins, the latter being different from the gluten of the endosperm. It consists of a minute plantlet with a cluster of leaves above, a radicle or embryo root below and, at the side next to the endosperm, a kind of sucker (Scutellum) for drinking in the sugar solution formed by the action of the enzyme diastase on the starch during germination. These details of structure will not, perhaps, be evident in the

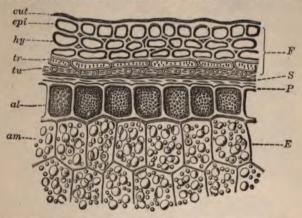
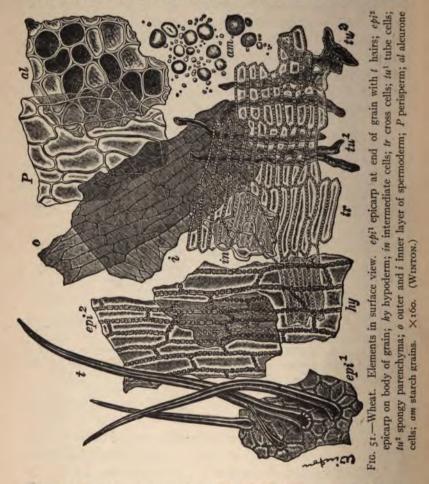


Fig. 50.—Wheat. Cross section through bran coats and outer endosperm. F pericarp consists of cut cuticle, epi epicarp, hy hypoderm, tr cross cells, and tu tube cells; S spermoderm consists of two brown layers; P perisperm; E endosperm consists of al aleurone cells and am starch cells. ×160. (MOELLER-TSCHIRCH.)

student's section, but he will note the absence of starch a demonstrated by the iodine test.

Histology. Fig. 50 shows the outer part of a cross section through the center of a wheat kernel magnified 160 diameters. Such a section is cut with a plano-concave razor or a mechanical section-cutter known as a microtome, after softening the kernel by soaking in water. As it requires considerable practice to secure good cross sections of grains and seeds, the student in the limited time allowed for this course should depend either on permanent mounts or else on the illustrations.

F is the fruit coat or *Pericarp*, consisting of three layers, not including the cuticle which is not cellular; S is the seed coat or *Spermoderm*, corresponding to the skin of an almond or bean;



P is the Perisperm or remains of the body of the ovule; E is the Endosperm, consisting of Aleurone Cells (al) and Starch Cells (am).

While cross sections are of great value for a scientific study of a vegetable product, especially in deciding as to the number WHEAT 97

and arrangement of the layers, they are neither so interesting nor so valuable in identification as surface preparations, that is, the layers removed by scraping the kernel, previously soaked for a time in water, with a penknife. In ground products cross sections are seldom found, the layers being largely in surface sections and, therefore, are seen through the microscope in surface view. Fig. 51 shows the successive layers of the wheat kernel in surface view, although not all of these are readily found or are of value in identification. The student should find all the important layers in water mounts of his own preparation.

The outer layer (Epicarp) consists for the most part of

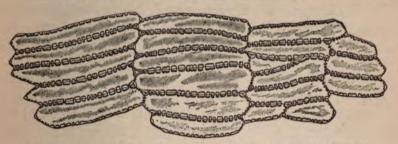


Fig. 52.—Wheat. Surface view of cross cells. X300. (K. B. Winton.)

elongated, distinctly beaded cells, so arranged as to "break joints" (Fig. 51 epi); at the apex of the kernel, however, the cells are polygonal and from among them arise the hairs of the beard. The Hairs (t) are broad at the base, pointed at the end, and have a distinct cavity the breadth of which is less than the thickness of the walls. They seldom, if ever, exceed  $1000\mu$  in length. A second and often a third layer of elongated cells, practically the same as those of the epicarp, are also present. The next layer (tr) consists of Cross Cells, so-called because they cross those of the preceding layers at right angles. Fig. 52 shows a group of cross cells more highly magnified. The cross cells are highly characteristic because they are arranged side by side in rows and, therefore, do not break joints. It should also be noted that both the thick side walls and the thin end

walls of the cells are distinctly beaded, whereas in rye the side walls are indistinctly beaded and the end walls are often swollen.

The curious cells of the *Intermediate Layer* (in) are not likely to be encountered and would not be mentioned were it not that the beginner often runs across the unusual. The *Tube Cells* (tu) which occur in all the cereals are remarkable in that they do not form a continuous layer, but occur isolated or only here and there in contact with one another. Such a formation is known as *Spongy Parenchyma*, and the spaces between such cells as *Intercellular Spaces*. In cross section (Fig. 50 tu), the tube cells appear as rings.

All the layers thus far enumerated, together forming a thin skin, make up the fruit coat or *Pericarp* corresponding to the flesh and stone (excluding the kernel) of the peach or the pod of the pea. None of the cells contains visible contents.

The two crossing layers of the seed coat or *Spermoderm* (Fig. 51, i and o), are still thinner, the cell walls appearing like mere lines. Were it not for their brown color they would be hardly noticeable. The *Perisperm* (P), is not evident without special treatment.

The Aleurone Cells (al) forming the outer layer of the endosperm, although conspicuous because of their thick walls and abundant contents of proteins and fat, are of little value in identification, as they occur in all the cereals and some other grains.

By far the greater part of the kernel consists of the thin-walled Starch Cells, also known as flour cells. These contain the Starch Grains (am), which we have already studied (p. 89, Fig. 43), imbedded in two proteins, Gliadin and Glutinin. These latter form with water Gluten, which gives wheat flour its peculiar dough-making properties and contributes so markedly to its nutritive value. The gluten, being a colloid, is not visible in a water mount except on special treatment.

The large lenticular starch grains are characteristic not of wheat alone, but of the group wheat, rye, and barley. The RYE 99

experienced microscopist can distinguish the three from one another by the size of these grains. In rye the grains often exceed  $50\mu$  in diameter, in wheat they practically never reach  $50\mu$ , while in barley they seldom exceed  $35\mu$ .

Characteristic Elements. (1) The hairs (Fig. 51, t; Fig. 54, T), distinguished with difficulty from rye hairs but readily from oat hairs (Fig. 54, A), by their shorter length (less than  $1000\mu$ ) and broad base; (2) the cross cells (Fig. 52), distinguished from rye cross cells (Fig. 53), by their more distinct beads and their thin beaded (not swollen) end walls; (3) the large lenticular starch

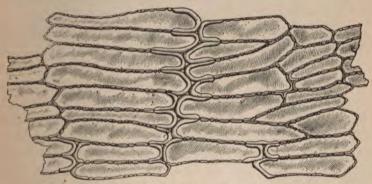


Fig. 53.—Rye. Surface view of cross cells. X300. (WINTON.)

grains (Fig. 43), not exceeding  $50\mu$  (distinction from rye), but often exceeding  $35\mu$  (distinction from barley).

\*Rye. The structure is throughout analogous to that of wheat. Study the layers noting the distinctions given in the foregoing paragraph. The difference in the Cross Cells (Figs. 52 and 53) serves to distinguish rye bran and other products containing the bran from the corresponding products of wheat. The difference in the size of the starch grains and the difference in the fragments of cross cells, obtained by special treatment to remove the starch, enable a skilled microscopist to distinguish rye flour from wheat flour. A more certain and often the only ready means of distinction is the following test:

Bamihl Test. This test, as modified by the author, consists

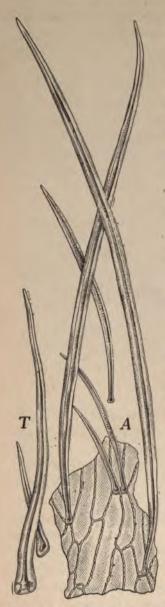


Fig. 54.—Hairs from wheat (T) and Oats (A). ×160. (WINTON.)

in mounting 1.5 milligrams of the flour in a drop of water containing in each liter 0.2 gram of watersoluble eosin. Before releasing the cover glass move it back and forth over the liquid, taking care that none of the flour escapes from beneath it. By this treatment the gluten of wheat flour and of a mixture containing a considerable part of wheat flour forms into rolls which greedily absorb the red dye and are readily seen with the naked eye. Rye flour, since it contains no appreciable amount of gluten, does not yield gluten rolls sufficient to be visible with the naked eye.

\*Oats. Both common barley and oats are known as chaffy cereals to distinguish them from naked cereals, such as common wheat, rye, and maize. It should be noted, however, that there are naked varieties of barley and chaffy species of wheat, although they are not of so common occurrence. The Chaff which closely invests the kernel is strongly silicified, as is also true of the stems or stalks of all cereals. The kernel after removal of the chaff is more slender than wheat or rye.

Histology. The structure of the chaff of oats and barley is highly interesting and of special importance to the food analyst, but need not be taken up in this short course.

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Suffice it to say that the two can be readily distinguished under the microscope.

None of the layers of the kernel up to the aleurone layer is at all conspicuous. The *Hairs* of the beard, however, are both striking and characteristic (Fig. 54, A). They often reach 2000µ in length and are, therefore, twice as long as wheat hairs. They taper not only toward the apex but also toward the base, which is so narrow as to appear almost pointed. The base of wheat hairs is broad.

The aleurone layer is striking, but not appreciably different from the corresponding layer of other cereals.

The Starch Grains (Fig. 44) of the starch or flour cells resemble those of rice, but are unlike those of any other cereal. They are small (seldom over 10µ), polygonal, and occur in rounded aggregates of from 2 to 100 individuals. As rice does not have

hairs, at least on the kernel freed from the chaff, these furnish a ready means of distinguishing oat from rice products. From all other cereal products the starch grains as well as the hairs are valuable means of distinction.

Characteristic Elements. (1) The hairs narrowed at the base (Fig. 54, A); (2) the polygonal starch grains in aggregates (Fig. 44).

\*Corn (Maize). A longitudinal section of the Indian corn or maize kernel (Fig. 55) shows very strikingly the division into oily embryo and starchy endosperm. The plantlet of the

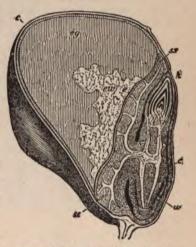


Fig. 55.—Corn. Longitudinal section.

c pericarp; eg horny and ew floury endosperm; sc and ss scutellum of embyro; k plumule; w primary root.

×6. (Sachs.)

embryo (at the right), has a distinct *Plumule* or group of leaves at the top (k) and a *Radicle* or embryonic roet below (w)

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At the center it is connected with the Scutellum (ss and sc), which draws the dissolved starch from the endosperm during sprouting. The Endosperm is partly floury (ew) and partly

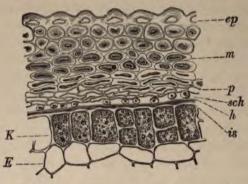


Fig. 56.—Corn. Cross section of bran coats and outer endosperm. Pericarp consists of ep epicarp; m mesocarp, p spongy parenchyma, and sch tube cells; h spermoderm; is perisperm; endosperm consists of K aleurone cells and E starch cells. ×160. (MOELLER.)

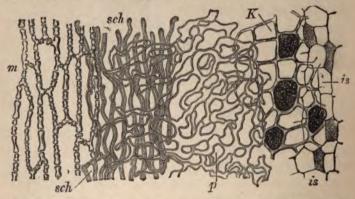


Fig. 57.—Corn. Bran coats in surface view. m mesocarp; sch tube cells; p spongy parenchyma; is perisperm; K aleurone layer. ×160. (MOELLER.)

horny (eg) the latter condition being due to the protein Zein in which the starch grains are embedded. The bran coats (c), including the aleurone layer, surround the endosperm.

Histology. The bran coats are shown in cross section and surface view in Figs. 56 and 57. The cells of the Outer Layers

(ep and m) in surface view remind us of the outer layers of wheat, but they are not so distinct, owing partly to the several layers which do not readily separate, and hairs are absent. The appearance of these layers in the yellow, white, or red skin will soon be learned by experience. The Tube Cells (sch), the loose or Spongy Parenchyma (p), and the Aleurone Cells (K) are not of special value in identification. The highly characteristic Starch Grains (Fig. 45) distinguish corn from all other economic products excepting the sorghums, which are not commonly milled. They range from 15 to 35µ in diameter and have a very distinct

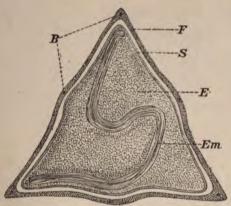


Fig. 58.—Buckwheat. Cross section. F pericarp with B bundles; S spermoderm; E endosperm; Em embryo. ×16. (Winton.)

hilum. In the horny endosperm most of the grains are polygonal; in the floury endosperm most of them are rounded.

Characteristic Elements. (1) The outer bran coats consisting of several layers of beaded cells; (2) the polygonal starch grains (Fig. 45) up to  $35\mu$  in diameter, with distinct hilum. Hairs are absent on the kernel although present in the chaff which, for the most part, remains with the cob in shelling.

\*Buckwheat. Although not a true cereal, buckwheat yields flour and by-products that are put to the same use as those of the cereals. The triangular grain is a dry fruit. Unlike the

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cereals the black hull or *Pericarp* is readily removed from the seed. The *Spermoderm* or seed coat is thin and papery of a brown or green-brown color. The *Embryo* is embedded in the *Endosperm* and so folded as to appear in cross section under a lens S-shaped (Fig. 58).

Histology. The brown elements of the black hulls need not be studied. The tissues of the seed coat are shown in Fig. 59. Especially noteworthy are the wavy-walled cells of the Outer

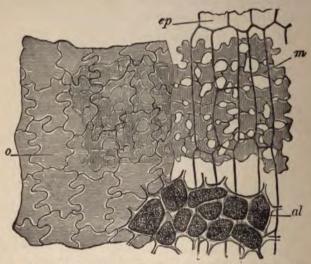


Fig. 59.—Buckwheat. Bran coats in surface view. Spermoderm consists of o outer epidermis, m spongy parenchyma, and ep inner epidermis; al aleurone cells. ×300. (MOELLER.)

Epidermis (o) which, if indistinct in a water mount, are brought out clearly by drawing a small drop of 5 per cent sodium hydroxide under the cover glass. The Spongy Parenchyma (m), with greenish or brownish cell contents, is also worthy of notice. Aleurone Cells like those of the cereals are present. Buckwheat Starch (Fig. 60), is slightly larger than oat starch, ranging up to over  $15\mu$  in diameter, but the grains are not so sharply polygonal and, although often united to form rod-shaped bodies, do not occur in rounded aggregates.

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(1) The wavy-walled cells of the Characteristic Elements. epidermis (Fig. 50, 0) and (2) the spongy parenchyma (m) usually suffice for identification. The absence of rounded aggre-

gates distinguishes the starch from oat and rice starch.

\*Peas. Beans and peas are true seeds. They consist of an outer skin or Spermoderm and an Embryo with large Cotyledons containing reserve starch. No endosperm is present in the mature seed, the food for the young plantlet having been



Fig. 60.—Buckwheat Starch. X300. (MOELLER.)

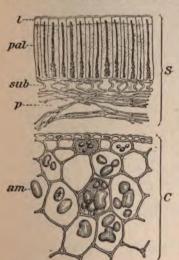


Fig. 61.—Pea. Outer layers in cross section. S spermoderm consists of pal palisade cells with I light line, sub column cells, and p parenchyma. C cotyledon with am starch cells. X160. (WINTON.)

eaten, but not digested as it were, during its development. Histology. Fig. 61 shows a cross section of the seed coat or spermoderm and cotyledon. The outer layer of the seed coat consists of high (60-100µ) but narrow cells forming a so-called Palisade Layer (pal). The cavity of these cells is narrow except at the base, where it is somewhat broadened. A curious "Light Line" (1) follows just within the outer surface of the layer. The next layer is of cells shaped like columns or hour glasses (sub). Both the palisade cells and Column Cells are isolated by heating a fragment of the skin, mounted in 5 per cent sodium hydroxide solution, and gently pressing the cover glass. After this treatment the palisade cells fall down on their sides while the

column cells assume various positions. The relative height of the cells of the two layers aids in distinguishing the different legumes. In beans each of the cells, corresponding to the column cells of peas, contains a beautiful crystal of calcium oxalate. The *Starch Grains* (am) of the pea are ellipsoidal, irregularly swollen, or bean shaped, varying in length up to  $40\mu$ . The hilum is elongated, but not so distinct as in beans or lentils. Many legumes contain starch, some, such as the soy bean and lupine, do not.

Characteristic Elements. (1) The palisade cells 60 to 100 $\mu$  high (Fig. 61, pal); (2) hour-glass or column cells up to

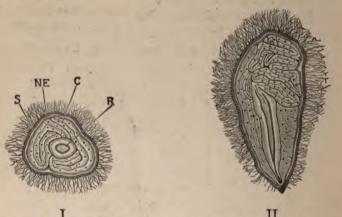


Fig. 62.—Cotton Seed. I cross section. II longitudinal section. S spermoderm; NE perisperm and endosperm; C cotyledons; R radicle. ×4. (Winton.)

20μ high (sub); (3) irregularly ellipsoidal starch grains up to 40μ (am).

\*Cotton Seed. A considerable number of economic seeds are characterized by the presence of oil instead of starch as reserve material. These "oil seeds" yield by pressure or extraction commercial oils, such as cotton seed, linseed, rape, sesame, cocoanut, palm, hemp seed, and poppy seed, which are used for foods, drugs, and various technical purposes, while the residual cake is commonly utilized for feeding cattle. Oil cakes contain a considerable amount of fatty oil which it is impracticable to remove and very high percentages of pro-

tein, because of which they are known as "concentrated feeds."

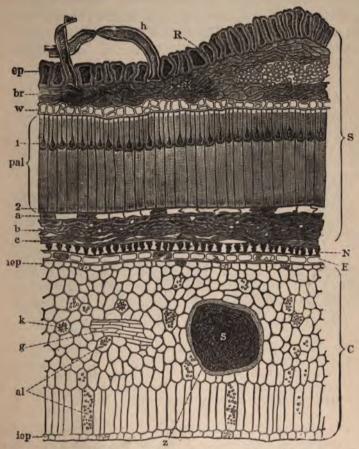


Fig. 63.—Cotton Seed. Cross section. S Spermoderm consists of ep epidermis with h hair, br outer brown coat with R raphe, w colorless cells, pal palisade cells, and a, b, c layers of inner brown coat; N perisperm; E endosperm; C cotyledor with aep outer and iep inner epidermis; s resin cavity surrounded by z mucilag cells; al aleurone grains; k crystal cells; g procambium bundle. ×160. (WINTON.)

Cotton seed, like the pea, contains its reserve material in its Cotyledons, but the starch is replaced by oil. A seed cut in

half with a jack-knife shows the thick black hull, seed coat, or spermoderm, and the gray-yellow much-folded cotyledons with minute resin cavities appearing as minute black spots (Fig. 62).

Histology. Fig. 63 shows a cross section through the seed coat and cotyledon and Fig. 64, the elements in surface view. Two layers are highly characteristic, viz., the Outer Epidermis (ep) of the seed coat and the Palisade Layer (pal).

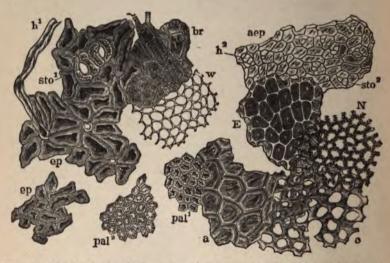


Fig. 64.—Cotton Seed. Surface view of outer layers. *ep* epidermis of spermoderm with  $h^1$  hair and  $sto^1$  stoma; br outer brown cells; w colorless cells;  $pal^1$  and  $pal^2$  palisade cells (see Fig. 63); a, b, c layers of inner brown coat of spermoderm; N perisperm; E endosperm; aep outer epidermis of cotyledon with  $h^2$  multicellular hair and  $sto^2$  stoma. ×160. (WINTON.)

The epidermal cells (Figs. 63 and 64, ep), obtained for study by scraping the outer surface of the seed, are of irregular shape with dark contents. Among them are the bases of the *Hairs* (Fig. 63, h; Fig. 64,  $h^1$ ) which remain attached to the seed after removing the major part by ginning. These hairs, the cotton fiber of commerce, are strap shaped, more or less twisted, and have a broad cavity or lumen.

The palisade cells (Fig. 63, pal) can be secured in suitable form for examination either by cutting thin cross sections of

the hull, using a section razor or a Gillette razor blade, or else by scraping the hull in a plane at right angles to the surface. They are remarkable for their great height  $(150\mu)$  and their division into an outer part of pure cellulose with a distinct cavity, about  $50\mu$  from the end, and an inner lignified part with no evident cavity.

The other layers of the seed coat are of no especial interest. The *Endosperm* (E) is reduced to a single layer of cells resembling the aleurone cells of the cereals in form and contents. The *Perisperm* (N), or remains of the body of the ovule, also consists of but one cell layer. The cell walls are curiously fringed. Since both endosperm and perisperm together form only a thin colorless coat neither tissue is prominent.

The bulk of the seed consists of the starch-free oily *Embryo*. In addition to Oil, which has no structure, Aleurone Grains (al) and occasional rosette crystals of Calcium Oxalate (k) are present. The aleurone grains are only 2 to  $5\mu$  in diameter and can be clearly seen only after removal of the fat from a section with a solvent such as ether and mounting in glycerine or else mounting directly in olive oil. As this involves considerable labor and with rather unsatisfactory results, the student may well reserve his study of aleurone grains until he examines flax seed in which they are large and quite distinct. The Resin Cavities (Fig. 63, s), contain a secretion which dissolves in strong sulphuric acid, the solution being blood-red.

In preparing and examining a mount in sulphuric acid take every possible precaution not to get any of the acid on the objectives or other parts of the microscope. Use only the low power objective and be sure it does not come in contact with the cover glass.

Characteristic Elements. The microscopic elements which serve for identification of cotton seed meal are (1) the epidermal cells of the hull, with yellow walls and dark contents (Fig. 64, ep); (2) the palisade cells (Fig. 63, pal); and (3) the resin cavities (s), the contents of which dissolve in sulphuric acid to a blood-red liquid.

\*Fiax Seed or Linseed. In this oil seed part of the reserve material is in the *Endosperm* (Fig. 65, E), and part in the *Embryo* (C). Figs. 66 and 67 show the seed coat and endosperm in cross section and surface view.

The Epidermis (Fig. 67, ep 1) of the seed coat consists of

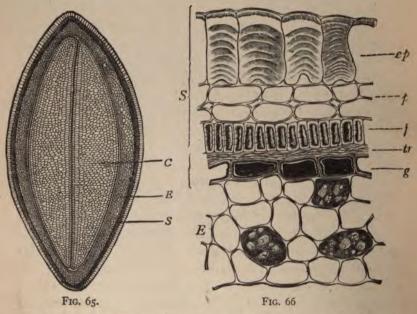


Fig. 65.—Linseed in Cross section. S spermoderm or seed coat; E endosperm; C cotyledons. (Moeller.)

Fig. 66.—Linseed. Cross section of S spermoderm and E endosperm. ep outer epidermis; p round cells; f fiber layer; tr cross cells; g pigment cells. (MOELLER.)

transparent, glassy cells. More readily found are the longitudinally arranged Fibers (f) with thick walls and ragged cavity, the thin-walled Cross Cells (tr), and the Round Cells (r). Often these three layers may be seen in the same fragment of the hull, by careful focusing. Equally striking are the more or less square Tannin Cells (pig). These have indistinctly beaded walls and brown contents. It should be noted that none of

the cells is perfectly square, but rather five or six sided with one or two of the walls much reduced in length. A perfectly square vegetable cell is physiologically as impossible as a square honey-comb cell.

A razor section of the seed mounted in olive oil, or some other

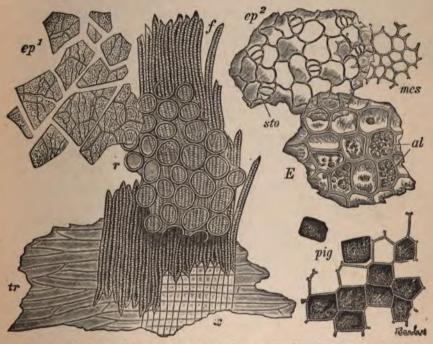


Fig. 67.—Linseed. Elements in surface view.  $ep^1$  epidermis of spermoderm; r round cells; f fiber layer; x middle lamellæ of fiber layer; tr cross cells; pig pigment cells; E endosperm with al aleurone grains;  $ep^2$  epidermis of cotyledon with sto immature stoma; mes mesophyl.  $\times 300$ . (K. B. Winton.)

fatty oil, or else in turpentine or glycerine (one of which will serve for the class) shows the large Aleurone Grains, of the endosperm and embryo (Fig. 66, E). They range up to  $20\mu$  in length. Aleurone grains are not, like starch grains, homogeneous in chemical composition. Each grain consists of a ground substance in which are usually embedded one or more Crystalloids

or protein crystals, one or more *Globoids* (compounds of lime and magnesia with phosphoric acid and an organic acid), and often crystals or crystal rosettes of *Calcium Oxalate*. In the aleurone grains of linseed only one globoid and one indistinct crystalloid are present.

Characteristic Elements. (1) Fragments consisting of fibers (Fig. 67, f), cross cells (tr), and round cells (r); (2) nearly square tannin cells with brown or yellow contents (pig).

\*Black Pepper. The chief characters of four of the principal spices—black pepper, cayenne pepper, cinnamon, and ginger—can be observed in a single exercise; the detailed structure, which could scarcely be mastered in a week, would for our purpose be of little more value.

Black pepper is the dried immature berry of a vine growing

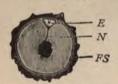


Fig. 68.—Black Pepper.

Longitudinal section of fruit. E endosperm; N perisperm; FS pericarp and spermoderm. × 3.

(MOELLER.)

in the Orient. White pepper is the mature berry from which the hull has been removed. The reserve material is in the form of starch and is contained, not in the endosperm as in wheat or in the embryo as in the pea, but in the Perisperm (Fig. 68, N), the robustly developed body of the ovule which in most plants largely disappears on ripening.

Histology. Fig. 69 shows the elements of ground black pepper. Just below the

Epicarp or epidermis of the berry is a layer (ast) consisting largely of Stone Cells, a kind of cell with thick lignified walls and branching cavities, widely distributed through the vegetable kingdom. The small but hard granules encountered in eating a pear or quince are groups of stone cells and the tough character of raspberry and strawberry "seeds" is due to a protective stone cell layer. Stone cells make up the bulk of nut shells (cocoanut, walnut, etc.), fruit stones exclusive of the kernel (peach, olive, etc.), the woody part of maize cobs, and various hardened tissues, but not of true wood. Stone cells of different products vary in form and size and in the thickness

and color of the walls and the nature of the cell contents. Because of these differences the trained microscopist can detect in ground pepper the presence of adulterants such as ground cocoanut shells and ground olive stones, which were formerly added in large quantities.

A second layer of stone cells (ist) occurs in the inner portion

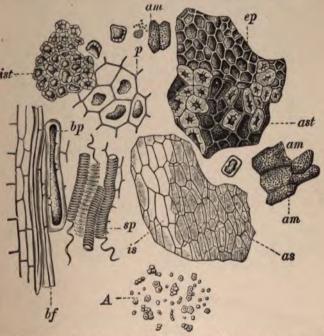


Fig. 69.—Black Pepper. Elements of powder. ep epicarp; ast hypodermal stone cells; bf bast fibers; bp bast sclerenchyma; sp vessels; p oil cells; ist endocarp; is and as layers of spermoderm; am starch masses. ×160. A starch grains, × 600. (MOELLER.)

of the hull. This portion is not removed in decortication, hence the stone cells occur in white as well as black pepper. As only the inner and side walls are thickened they are known as *Beaker Cells*. This character is not evident in surface view of the stonecell groups, but only when individual cells become detached as shown at the right of the group shown in the figure.

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The bulk of the pepper corn consists of a mass of Starch Cells. The ground product contains groups of cells with contents intact (am), also the starch separated from the cells as individual grains or groups of grains (A). The individual grains are among the smallest found in economic products, being usually 2 to 4  $\mu$  in diameter and never exceeding  $6\mu$ . Needle-shaped crystals of *Piperine* are often evident in ground pepper.

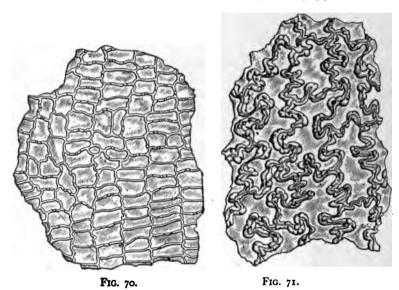


Fig. 70.—Cayenne Pepper. Epicarp in surface view. ×160. (K. B. Winton.)

Fig. 71.—Cayenne Pepper. Epidermis of seed in surface view. ×160. (K. B. Winton.)

Characteristic Elements. (1) The stone cells (Fig. 69, ast) of the outer layer; (2) the beaker-shaped stone cells (ist); (3) the starch cells (am); (4) the liberated starch (A) with minute grains.

\*Cayenne Pepper. The highly pungent chillies, or fruits of a small podded species of Capsicum grown in Africa, are known in commerce as cayenne pepper or cayenne. The mild fruits of a large podded variety of the garden pepper grown in Hungary yield paprika.

Histology. Both kinds of red pepper are non-starchy and

contain in the pod tissues drops of *Oil* which take on an orangered coloring matter also formed in the cells. Mounted in concentrated sulphuric acid the oil drops become indigo blue. The tissue characteristic of cayenne alone is the *Epicarp*, or outer epidermis of the pod, consisting of more or less rectangular cells with wavy walls (Fig. 70).

The *Epidermis* of the seed consists of remarkable cells with curious, wrinkled walls resembling the convolutions of the intestines (Fig. 71). As seen in surface view these cells are much alike in cayenne and paprika, but no such cells occur in any other common food product. The seed tissues containing oil and aleurone grains are not remarkable.

Characteristic Elements. (1) The more or less rectangular cells of the epicarp with wavy walls (Fig. 70); (2) the orange-red oil drops becoming indigo blue with sulphuric acid; (3) the intestine cells of the seed (Fig. 71).

\*Cinnamon. The moderately thick bark which in import trade is designated cassia when ground is known to the house-wife as cinnamon. True or Ceylon cinnamon is a very thin bark used chiefly in medicine.

Histology. The scientific study of barks involves a knowledge of the so-called Fibro-vascular Bundles, forming the conductive system of plants. Such a study, although highly interesting, would carry us beyond the limits of our work. For diagnostic purposes we need consider only such elements as are most conspicuous in the powdered material, namely the bast fibers, the stone cells, the cork cells, and the starch grains.

The Bast Fibers (Fig. 72, bf) resemble stone cells in general structure and chemical composition, but are elongated, pointed at both ends, and have a smooth (not branching) cavity. The flax fibers used in making linen fabrics are bast fibers.

The Stone Cells are either thickened on all sides (st) or only on one side (stp). Cork Cells (P) are present if the bark has not been deprived of its outer layers by scraping. The cork of commerce is the highly developed cork layer of an oak grown in

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Spain. Owing to the infiltrated *Suberin*, cork cells repel water and form a protective coat for the tree or plant. All the preceding elements are best seen after treating a water mount with sodium hydroxide; the starch, however, must be examined in the water mount.

The Starch Grains (B) range usually from 10 to  $20\mu$  in diameter, and occur mostly in aggregates of 2 to 4 individuals. They

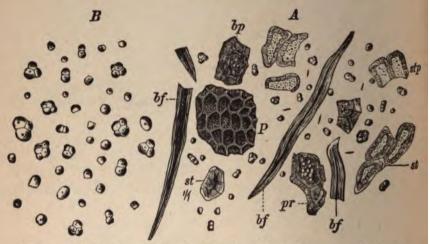


Fig. 72.—Cinnamon. A elements of powder: bf bast fibers; st and stp stone cells; pr and bp parenchyma; P cork. ×160. B starch grains, ×600 (MOELLER.)

have rounded or flat sides according to their location in the aggregates. A distinct hilum is evident.

Characteristic Elements. (1) Bast fibers (Fig. 72, bf); (2) stone cells (st, stp); (3) starch grains (B), 10 to 20 $\mu$ , with distinct hilum, occurring mostly in aggregates of 2 to 4.

\*Ginger. The dried underground stem or root of the ginger plant comes into the market simply washed or else scraped. A coating of chalk is added to some varieties the product thus limed, being, it is claimed, less susceptible to the attacks of insects.

Histology. The rootstock consists in large part of paren-

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chyma cells filled with Starch Grains which are characteristic because of the rounded angle at one end (Fig. 73, am). Most of

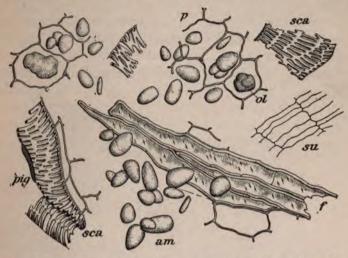


Fig. 73.—Ginger. Elements of powder. p parenchyma with starch grains and ol oil masses; am starch grains; f bast fibers; sca vessels; pig pigment; su cork. ×160. (K. B. WINTON.)

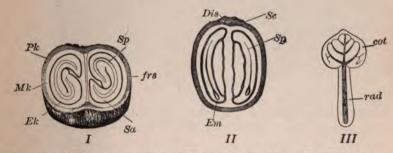


Fig. 74.—Coffee. I cross section of berry, natural size. Pk outer pericarp;
Mk endocarp; Ek spermoderm; Sa hard endosperm; Sp soft endosperm.
II longitudinal section of berry, natural size; Dis bordered disc; Se remains of sepals; Em embryo. III embryo, enlarged: cot cotyledon; rad radicle.
(TSCHIRCH and OESTERLE.)

the grains are egg shaped and 20 to  $30\mu$  long, although smaller and larger grains (up to  $50\mu$ ) occur sparingly. The fibrous

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material of the rootstock contains Vessels (sca) with reticulated thickenings and Bast Fibers (f) with rather thin walls and broad cavities.

Characteristic Elements. (1) Starch grains with rounded angle at one end (am); (2) reticulated vessels (sca); (3) bast fibers (f) with thin walls and broad cavity.

\*Coffee. In a laboratory period the student can not only

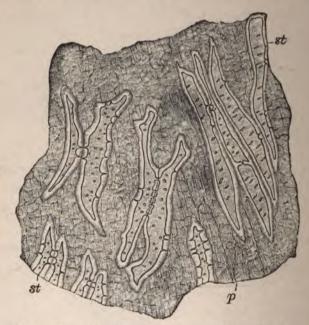


Fig. 75.—Coffee. Spermoderm in surface view. st stone cells; p parenchyma ×160. (MOELLER.)

learn the general structure of coffee and cocoa, but also the use of the section razor in studying these products. Fig. 74, I and II, shows cross and longitudinal sections of a coffee berry or "bean" natural size. The shelled bean consists of the hard endosperm in which is embedded a minute embryo (III).

Histology. The papery fragments of the spermoderm or seed coat, which will be found in the cleft of a coffee bean,

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should first be examined. Scattered here and there over the skin are remarkable *Stone Cells* of various shapes with porous walls and broad cavities (Fig. 75, st). Often two or more of the cells are in groups.

The structure of the endosperm is best seen in a cross section. Such sections can be secured by holding a coffee bean, which has been softened by soaking or boiling in water, between the thumb and first finger of the left hand and cutting the thinnest possible shavings with a section razor or Gillette blade held with the right hand. Considerable experience is required for cutting satisfactory sections of certain seeds, but a little practice should enable the student to prepare sections of the coffee bean thin enough to show the general character of the cells.

It will be noted that the cell walls (Fig. 76) are not only thick,

but have a beaded appearance due to the pits or pores which pierce them, thus furnishing communication from one cell to another. The thickened cell walls constitute the chief reserve material which, instead of being in the form of starch or oil, is in the form of cellulose or related carbohydrates. Sections can be cut of small

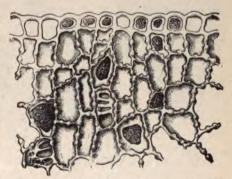


Fig. 76.—Coffee. Cross section of outer layers of endosperm showing knotty thickenings of cell walls. ×160. (MOELLER.)

fragments if held between flat pieces of cork. In this manner ground coffee can be distinguished from common adulterants and substitutes, such as peas and wheat. As a preliminary test, a teaspoonful of the sample should be stirred in a glass of cold water. Peas, cereals, and chicory will sink at once, while coffee floats. Chicory quickly imparts to the water a dark color.

Characteristic Elements. (1) Stone cells of the papery seed

coat (Fig. 75, st); (2) reserve cellulose in the form of knotty thickened or beaded walls of the endosperm (Fig. 76).

\*Cocoa Bean. The seed of the cocoa or cacao tree, known as the cocoa bean (Fig. 77), consists of a leathery hull or shell and an embryo with folded but fleshy cotyledons containing the reserve material, which is partly starchy and partly oily, the latter predominating. Cocoa beans yield after roasting, shelling, and grinding, the chocolate of commerce. The heat

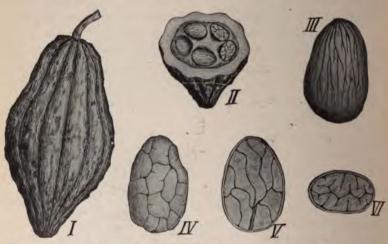


Fig. 77.—Cocoa. I entire fruit, X<sup>1</sup>/<sub>4</sub>; II fruit in cross section. III seed (cocoa bean), natural size; IV seed deprived of spermoderm; V seed in longitudinal section, showing radicle (germ); VI seed in cross section. (WINTON.)

of the grinding melts the fat or cocoa butter, and as a consequence, the product which runs from the mill is a thick paste, which hardens on cooling to a waxy mass known as plain or bitter chocolate or chocolate liquor.

Cocoa is the cake remaining after pressing out about half of the fat, reduced to a powder.

Histology. Fragments scraped from the shell of a cocoa bean should first be examined. In these will be seen numerous spirals, like spiral springs, which are the thickenings of Spiral Vessels. These give rigidity to the vessels, serving the same

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purpose as the spiral iron wires in the flexible pipes for gas drop lamps. Spiral vessels occur also, but sparingly, in black pepper (Fig. 69, sp).

Cross sections should then be cut of the cotyledons without soaking, the high percentage of fat being sufficient to make them soft, yet firm. These sections may be examined directly in water, but the fat will greatly interfere with the observation. It is better to remove the fat from the section, previous to

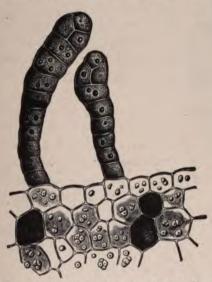


Fig. 78.—Cocoa. Cross section of outer portion of cotyledon, showing hairs and starch parenchyma. (MOELLER.)

mounting in water, by treating with several successive portions of ether or chloroform in a watch-glass, or else to mount in turpentine. In either case the starch grains will be evident, but the water mount of the extracted sections has the advantage that the iodine test can be applied.

Cocoa Starch (Fig. 78) resembles that of cinnamon. The grains are  $4-12\mu$  in diameter, and occur in aggregates of two to four individuals. Because of their grouping into aggregates, they often have both rounded and angular outlines.

Characteristic Elements. (1) Numerous spiral vessels in the shells; (2) starch grains  $4-12\mu$  in diameter with evident hilum, occurring in small aggregates.

\*Tea. Both black and green tea consist of the dried leaves of Thea Chinensis, the difference in color being due to the



Fig. 79.—Tea. Leaf, nat-

process of drying. The leaves (Fig. 79), as may be seen by spreading out the moist residue after preparing the beverage, are rather thick, glossy on the upper surface, short-toothed, and veined in such a manner that the main veins branching off from the midrib form near the margin loops, connecting them with adjoining veins.

Histology. The structure of leaves bears an obvious relation to their function, namely, *Photosynthesis*, the formation of carbohydrate matter from carbon dioxide of the air and water of the soil in the sunlight through the agency of chlorophyl grains.

Fig. 80 is a cross section of a tea leaf through a *Stoma*. Beneath the stoma there is an air space surrounded by loosely arranged cells containing *Chlorophyl Grains*, and often crystals of *Calcium Oxalate*. It is here that photosynthesis takes place. A large curiously formed *Stone Cell* extends from one epidermis to the other. Other forms of stone cells are seen at the right (st).

A surface section of the lower epidermis (Fig. 81) shows several stomata (sp), also a Hair(h) bent near its base like a cane handle.

Characteristic Elements. All of the elements named may be found in the debris obtained by scraping a moist tea leaf. The curious stone cells and the hairs are characteristic.

\*Examination of Mixtures in Powder Form. Various mixtures should be made of the following: wheat flour, buckwheat

flour, maize flour, ground wheat bran, ground rye bran, ground oat meal, ground peas, cotton seed meal, linseed meal, ground

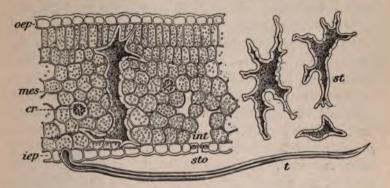


FIG. 80.—Tea. Cross section of leaf. *iep* lower epidermis with t hair and sto stoma; mes mesophyl with chlorophyl grains, large stone cell, and cr calcium oxalate rosette; int intercellular space; oep upper epidermis; st isolated stone cells. ×160. (WINTON.)

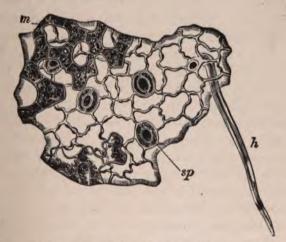


Fig. 81.—Tea. Leaf seen from below, showing epidermis, with h hair and sp stoma, and m mesophyl.  $\times 160$ . (Moeller.)

black pepper, ground cayenne pepper, ground ginger, ground cinnamon, and cocoa. Mixtures of more than two of the materials seem inadvisable, at least if only a day can be devoted to the work of identification. In order that the color may not furnish a clue to the ingredients, pigments such as yellow ochre, burnt sienna, and lampblack may be added to some of the mixtures. Only such mixtures should be prepared as correspond to products now or formerly on the market. Ground coffee, pure and mixed with roasted ground peas or roasted wheat, may also be used for practice material.

An examination should first be made of a water mount, before and after staining with iodine, to determine whether or not starch is present and, if so, the kind. A drop of sodium hydroxide can then be drawn under the cover glass and the tissues examined. Reference should be made to the paragraphs of the preceding descriptions, giving the "Characteristic Elements" of the products, in interpreting the results of the observations.

The following hints may be useful: If lenticular starch grains 30 to 50 and hairs with broad bases are found, wheat or rve is present. If further search discloses a considerable number of starch grains over 50 µ, these are doubtless from rve. In any event cross cells should be looked for, and the characters distinguishing wheat from rye noted. As the small starch grains of wheat and rye resemble buckwheat starch, search should be made for the tissues of the buckwheat seed coat, especially the wavy-walled cells and green-brown spongy parenchyma. Oat starch might also be confused with the small grains of wheat and rye or buckwheat starch, were it not for the presence of aggregates of numerous individuals; furthermore the long oat hairs with narrow, almost pointed, bases are characteristic. Bean-shaped starch grains with elongated hilum indicate peas (knowing other leguminous seeds to be absent) and large (up to 35µ) polygonal grains are evidence of maize. Pea hulls in the ground product will be indicated by the presence of isolated palisade cells and hour-glass cells. Cotton seed and linseed meals contain no starch, but the tissues of the seed coat leave no doubt as to their identity.

Of the spice starches, that of pepper is minute, of cinnamon

larger and in small aggregates, and of ginger large with a rounded angle at one end. Cayenne contains no starch but the orange oil drops, the epicarp, and intestine cells are characteristic. The stone cells of pepper and the bast fibers as well as the stone cells of cinnamon should be noted. Broad bast fibers and curious vessels are present in ginger. An added cereal or oil seed product may be looked for in spices, also wheat or maize flour in cocoa.

Roasted peas, or wheat if present in ground coffee, sink in cold water. The presence and characters of the starch and the elements of the hulls or bran furnish more specific information. Chicory also sinks in cold water-imparting a brown color.

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# CHAPTER VI

## SACCHARINE PRODUCTS

### SUGAR

Characters of Sucrose. Since, at the present time, the world's product of commercial sugar is obtained in about equal quantities from the sugar cane and the sugar beet, and furthermore, the same sugar is also produced by the sugar maple tree, the frequent use of the term cane sugar as a synonym for sucrose is confusing.

Sucrose is a disaccharide with the empirical formula  $C_{12}H_{22}O_{11}$ . It is dextrorotatory, that is, a water solution turns a ray of polarized light to the right. Treatment with 2.5 to 4 per cent hydrochloric acid at 69° C. for five to fifteen minutes or at 20° C. for one or two days inverts sucrose, causing it to split up into one molecule each of *Dextrose* (d-glucose) and *Levulose* (d-fructose) as shown by the following equation:

$$C_{12}H_{22}O_{11} + H_2O = C_6H_{12}O_6 + C_6H_{12}O_6$$

Although the two cleavage products have the same empirical formula, dextrose is an aldehyde (aldose) and dextrorotatory, while levulose is a ketone (ketose) and levorotatory. The fact that both dextrose and levulose reduce Fehling's copper solution, while sucrose does not, is explained by the hypothesis that the two radicles in the latter are so combined as to destroy the aldehyde group of the one and the ketone group of the other.

This difference in configuration is brought out strikingly by the following structural formulae of the three sugars:

The copper-reducing power of dextrose is slightly greater than that of levulose; on the other hand, the levorotatory power of levulose at ordinary temperatures is considerably more than the dextrorotatory power of dextrose, consequently *Invert Sugar*, the mixture of equal molecules of dextrose and levulose, is levorotatory.

The characters which have been briefly stated are the basis of the most important analytical methods used in sugar analysis. Sucrose is calculated from the figures obtained by polarization both before and after inversion, while dextrose, levulose, and invert sugar are commonly determined by copper reduction.

The Polariscope. The instrument used in sugar analysis known are Saccharimeter is graduated in terms of percental and do sing the solutions to definite volumes. A full consideration of the construction of the different types of saccharimeter and the optical principles involved would more than fill the pages of this volume. As is also true of the microscope, there is no more necessity of the student understanding fully the detailed construction or optics of the instrument, than for an artist to understand the anatomy, physiology, and optics of his own eyes. The following brief statements may add interest to the work.

Fig. 82, taken from Browne's Handbook of Sugar Analysis, is a diagram of the simplest form of polariscope. The light from the lamp L is polarized by the Nicol prism P, known as the polarizer, that is, the light vibrations which are normally in different planes are reduced to one plane. Another Nicol prism, A, known as the analyzer, is so arranged that it can be rotated about the longitudinal axis of the instrument. With

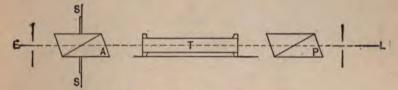


Fig. 82.—Diagram of a Simple Polariscope.

the sugar tube T empty, the light polarized by P, as seen by the eye at E, is not altered by A when the axes of the two prisms are parallel; when, however, the axes are crossed, the light is extinguished. If with crossed prisms, a sugar solution is placed in T, the ends being closed by circular glasses, the light is no longer extinguished, but passes through with greater or less intensity, dependent on the nature and amount of the sugar. On turning the analyzer so that the field is gain

black, the amount of rotation due to the sugar solution can be read on the circular scale S.

The chief difficulty with an instrument of this simple construction would be to determine the point where the field is

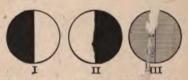


Fig. 83.—Double Field of Hal shadow Saccharimeter.

darkest. To obviate this defect a half-shadow device with a double-field is employed, whereby the left half of the field is dark and the right half is light when the analyzer is crossed with the left half of the field (Fig. 83, I), while the reverse is true when the analyzer is crossed with the right half of the field (Fig. 83, II). The zero point, when the tube is empty,

and the end point, when an analysis is being made, is shown by the exact correspondence of the two halves of the field (Fig. 83, III).

A further improvement is the double-wedge system, in which both analyzer and polarizer are fixed, and the end point is obtained by sliding one quartz wedge alongside of another until the thickness of the two is such that the quartz

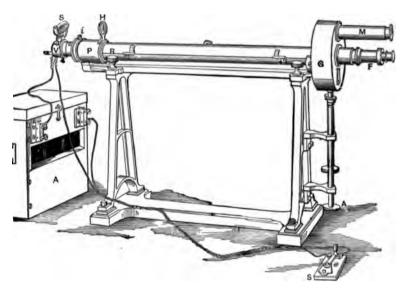


Fig. 84.—Double-wedge Soleil-Ventzke Saccharimeter with Bock Stand and Electric Lamp.

rotates the light to the same degree as the sugar solution. The reading is taken on the scale or scales with which the wedge device is provided. Fig. 84 shows a modern instrument with double-field and double-wedge device.

\*Polarization of Granulated Sugar before and after Inversion. The best granulated sugar is practically pure sucrose. It contains only traces of water, ash, and reducing sugars.

Weigh out in a nickel sugar dish (Fig. 85) the normal quan-

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tity of sugar for the type of saccharimeter used (26 grams for the Soleil-Ventzke saccharimeter), transfer through a funnel to a 100-cc. graduated flask, rinsing out any that may adhere to the funnel with water. Add enough water to dissolve the

sugar and make up to the mark and shake.

Direct Polarization. Remove the cap and cover-glass from one end of a 200-mm. observation tube (Fig. 86), fill with the sugar solution, slide the cover glass into place, and attach the cap.



Fig. 85.—Nickel Sugar-weighing Dish and Counterpoise.

If the tube has one end enlarged as shown in Fig. 86, it is not necessary to avoid an air bubble in the tube, as this will rise, when the tube is in a horizontal position, into the expanded part of the tube and thus be out of the line of vision; if, however, both ends are the same size (Fig. 87) the tube



Fig. 86.—Saccharimeter Tube with Enlarged End.



Fig. 87.—Saccharimeter Tube, Simple Form.

must be completely filled and the cover glass slid into place in such a manner as to exclude any air bubble.

Place the tube in the polariscope, light the lamp, adjust one scale at o, and move the other until the end point is reached. The reading should vary only slightly from +100.

Polarization after Inversion. Pipette 50 cc. of the sugar solution and 25 cc. of water into a 100-cc. graduated flask, add 5 cc. of concentrated hydrochloric acid, mix, place a ther-

mometer in the solution, and heat in a water bath at  $72^{\circ}$  to  $73^{\circ}$  C., so that the solution reaches  $69^{\circ}$  in  $2\frac{1}{2}$  to 5 minutes. Maintain at  $69^{\circ}$  C. for five minutes, remove the flask, cool rapidly to  $20^{\circ}$  C. under a stream of cold water, and dilute to  $100^{\circ}$  Cc.

Polarize in a 200-mm. tube as before inversion, but multiply the reading, which will be to the left (expressed by the minus sign), by 2 to compensate for the dilution from 50 cc. to 100 cc. Immediately after taking the reading, plunge a thermometer in the solution to determine the exact temperature. If the polarization is determined at exactly 20° C., the reading multiplied by 2 should vary only slightly from -32.7.

We are now in a position to understand the Herzfeld-Clerget formula, as follows:

$$S = \frac{100(a-b)}{142.66 - t/2},$$

in which S = per cent of sucrose, a = direct polarization (with + sign), b = invert polarization (with - sign), and t = the temperature.

a-b, being the algebraic difference, will be the sum of the direct (+) and invert (-) polarization. If the work has been carefully performed, the percentage of sugar by direct polarization, as well as by the above formula, will be practically 100.

In a case such as the preceding, where only sucrose is present, the inversion is not necessary for the correct result; if, however, another sugar, such as dextrose or invert sugar, is present, the result of the direct polarization is not the percentage of sucrose, but a meaningless resultant figure, whereas the Herzfeld-Clerget formula automatically corrects for the polarization of the foreign sugar or sugars and gives the true percentage of sucrose. This is readily understood when it is noted that both the reading a and the reading b consist in part of the same figure, namely the polarization of the foreign sugar. As this figure is introduced twice with opposite signs, its influence is eliminated from the equation, and the result due to the polarization of the sucrose alone is obtained.

### Molasses, Syrup, and Honey

Chemical Composition. Molasses and Sugar House Syrups are solutions of sucrose with some reducing sugars and various other impurities. Lead subacetate precipitates the bulk of the nonsaccharine impurities which give the product its dark color.

Commercial Glucose or Corn Syrup is prepared by the action of dilute hydrochloric acid on starch, the acid being subsequently neutralized by sodium carbonate with the formation of ordinary salt. Its solid matter consists of a mixture of maltose, dextrine, and dextrose, all of which are dextrorotatory. The product used for mixtures such as Karo Syrup, is known as 42° Baumé mixing syrup and polarizes from 162° to 175° on the sugar scale.

Honey consists essentially of the sucrose from the nectar of flowers inverted in the body of the honey bee. It is levorotatory. Hawaiian honey-dew honey and unimportant varieties made from the blossoms of certain trees are often dextrorotatory.

\*Determination of Sucrose in Molasses, Syrup, and Honey. Material for Laboratory Practice. Provide for practice material a good grade of molasses, the product known as Karo Syrup, or a similar mixture of about 90 parts of commercial glucose or corn syrup and 10 parts of cane syrup, and pure strained honey (not Hawaiian).

Method of Clarification. In order to polarize colored or turbid products, such as molasses, syrups, and honey, it is necessary first to clarify them, that is, to remove the larger part of the coloring matter and all of the suspended or colloidal impurities.

Weigh out in a nickel sugar dish normal quantities (26 grams) of the molasses and honey and a half normal quantity of the syrup. Transfer with the smallest possible amount of water to 100-cc, graduated flasks. To the molasses and syrup add with shaking lead subacetate solution (sp.gr. 1.25) in slight excess (5 to 10 cc.) and 1 cc. of alumina cream (aluminum

hydroxide suspended in water); to the honey add only alumina cream. Make up to the mark, shake, and filter through dry filters into dry flasks. Instead of the solution of lead subacetate the powdered dry salt, as recommended by Horne, may be added, after making up to 100 cc., until no further precipitation is noted. For the preparation of alumina cream a solution of aluminum sodium sulphate is precipitated with ammonia water, washed by decantation until sulphates are removed, and finally decanted down to the precipitate.

Direct Polarization. Polarize the filtrates as described for granulated sugar (p. 131). Note that the molasses and syrup are dextrorotatory, the syrup being strongly so, although only half the normal quantity was used, while the honey is levorotatory.

A little practice is required in manipulating the scales to distinguish right- and left-handed polarization. As different instruments have different mechanisms the student must depend upon the instructor or his own experience in such details.

Invert Polarization. Free portions of the solutions clarified with lead subacetate from lead by adding finely powdered potassium oxalate with shaking until no further precipitate forms. Filter through dry filters into dry flasks. The process of inversion may be conducted at 69° C. exactly as in the inversion of granulated sugar. However, in order to vary the manipulation and divide the work between two days, the inversion should be carried out at room temperature as follows: Place 50-cc. portions of the solutions (deleaded in the case of the molasses and the syrup) in 100-cc. flasks, add 5 cc. of concentrated hydrochloric acid, mix, and allow to stand overnight (at least twenty-four hours), at a temperature not below 20° C.

Dilute to 100 cc., polarize, and calculate sucrose by the Herzfeld-Clerget formula, as described for granulated sugar (p. 132). Note that the invert polarization of the molasses is to the left, while that of the Karo Syrup is still to the right, although less than before inversion. The polarization of the

honey is not greatly affected by the inversion. The calculated amount of sucrose in the molasses should be between 30 and 55 per cent, in the Karo Syrup about 10 per cent (or the amount declared on the label), and in the honey a very small amount, if any.

\*Calculation of the Total Solids in Molasses, Syrup, and Honey from the Refractive Index. The Abbé refractometer, being most commonly used in the examination of fats and oils, is described in Chapter VII. Determine the refractive index as described for oils (p. 149) and calculate the total solids by means of Geerlig's table (p. 224), correcting the results for temperature in accordance with the table on p. 225.

The drying of saccharine products in an ordinary water oven at 100 °C. is a slow operation. If levulose is present, the results are inaccurate, owing to the decomposition of that sugar. This error can be obviated by drying at 70°C. in a vacuum oven, but the apparatus is expensive and troublesome to maintain. All drying methods are tedious. The calculation from the refractive index is convenient and sufficiently accurate for practical purposes.

#### MAPLE PRODUCTS

Source. The sap of the sugar maple tree is rich in sucrose and contains in addition certain characteristic flavoring constituents. On evaporation Maple Syrup and Maple Sugar are obtained.

Because of the increased demand and consequent high price of maple products, cheaper saccharine products are mixed with them, thus diluting the flavor although not reducing the food value. Formerly glucose was used, but at the present time refined sugar syrup is preferred.

Analysis of Maple Products. As the sugar of the sugar cane, sugar beet, and maple tree are identical, determinations of sucrose are of no value in the examination of maple products with reference to the admixture of refined sugar. Dependence must therefore be placed on the character and amount

of minor constituents. Fortunately for the consumer, it is impracticable to add molasses or sugar-house syrup, as these have strong flavors that would conceal the delicate maple flavor. Being forced to use refined sugar, the detection of a considerable admixture is readily accomplished by determinations of Ash and Lead Number, the latter being a measure of the constituents precipitated by basic lead acetate. Refined sugar contains practically no ash and no constituents precipitated by lead salts.

#### FRUIT SYRUPS

\*Artificial Colors in Fruit Syrups. The coloring of foods with vegetable, animal, and coal-tar dyes has been a matter of much concern to food chemists. In certain foods such as fruit products the dyes used conceal inferiority or adulteration, while in other foods such as confectionery, the purpose is merely to please the eye.

Certain State laws allow only vegetable and animal colors. Most State food laws and the regulations of the Federal Food and Drugs Act permit the use not only of harmless vegetable and animal colors, but also certain coal-tar dyes provided their presence is declared on the label and they are not used to conceal inferiority.

Fruit syrups, especially those used for soda water, are often colored and may be used as representative products for laboratory practice.

\*Material for Laboratory Practice. The following should be provided for use of the class:

- 1. Pure Raspberry or Strawberry Syrup. If a product of known purity is not obtainable express the juice of the fruit, add sugar, and boil down to a syrupy consistency. Lacking the fresh fruit, jam may be used as the basis of the syrup.
- 2. Simple Syrup (prepared by treating granulated sugar with water in the proportion of 100 grams of the sugar to about 60 cc. of water) colored with cudbear.
  - 3. Simple Syrup colored with cochineal.

- 4. Simple Syrup colored with amaranth.
- 5. Simple Syrup colored with ponceau 3R.
- 6. Simple Syrup colored with erythrosin.
- 7. Simple Syrup colored with orange I.
- 8. Simple Syrup colored with naphthol yellow S.

The amounts of the artificial red dyes used in 2 to 6, inclusive, should be sufficient to impart to the syrups a red color of about the intensity of that of the raspberry syrup; the amount used in 7 and 8 should be sufficient to color the syrups bright orange and yellow shades. Federal regulations permit the addition to foods of all the dyes enumerated. In addition to the five coaltar colors (4 to 8, inclusive), three others (light green S. F., yellowish, indigo disulphoacid or indigo carmine, and tartrazine) may also be used.

Syrups 2 to 8, inclusive, will give the same results in dyeing tests as imitation fruit syrups flavored with fruit ethers and containing the colors named.

\*Arata's Wool Dyeing Test. Dilute 10 to 25 cc. of the syrup to 100 cc., add 10 cc. of a 10 per cent solution of potassium bisulphate and a piece of white woolen cloth (nun's veiling or albatross cloth), about 1 in. square, which has previously been heated to boiling in a 0.1 per cent solution of sodium hydroxide to remove any grease. Heat to boiling and boil for about fifteen minutes. Remove the wool, boil first with water, then with a solution of an alkali-free soap, and finally again with water. Dry and note the color.

The coal-tar colors will dye the cloth bright red, orange, or yellow shades, whereas the cudbear dyes it a dirty red color, the cochineal a pinkish color, and the natural color of the raspberry scarcely at all.

A few drops of ammonia added to small pieces of the dyed cloth in a watch-glass bring out the following colors: Cochineal, purplish; cudbear, violet; amaranth, brown; ponceau 3R, pink; erythrosin, pink; orange I, deep red; naphthol yellow S, yellow. Concentrated sulphuric acid added to other pieces develops the following colors: Cochineal, pink; cudbear, dirty color; Amar-

anth, violet; ponceau 3R, cherry red; erythrosin, orange; orange I, magenta; naphthol yellow S, decolorized.

The reactions thus obtained, although quite decisive for the small number of colors used in our samples, are not so satisfactory when the hundreds of possible colors are involved, especially when two or more colors are present in the same product.

Some natural fruit colors and various vegetable dyestuffs impart to the cloth a dirty color which gives indistinct color reactions. In such cases it is well to dissolve the color from the cloth by boiling with dilute ammonia water and repeat the dyeing test with a smaller piece of wool. While the coal-tar colors and some of vegetable origin dye the second piece of wool, fruit colors do not.

Sostegni and Carpentieri use a few drops of hydrochloric acid in place of the potassium bisulphate solution.

\*Robin's Test for Cochineal. Dilute 10 cc. of syrups 1 and 3 with an equal volume of water, add a few drops of hydrochloric acid, and shake in a separatory funnel with 25 cc. of amyl alcohol. Cochineal imparts to the amyl alcohol a yellowish color. Draw off the aqueous liquid and shake the amyl alcohol solution with several portions of water until neutral. To a portion of the amyl alcohol extract in a test-tube add a few drops of uranium acetate solution, shaking after each drop. If cochineal is present, the reagent acquires a beautiful emerald green color. To another portion add ammonia water. Cochineal, after this addition, gives a violet coloration.

Ammonia water added directly to the syrup colored with cochineal gives the violet coloration. The same reagent added to the syrups colored with cudbear or other lichen color becomes blue.

Mathewson has devised special tests for coal-tar colors which often give decisive results when the foregoing simple tests fail.

### CHAPTER VII

#### FATS AND OILS

Constitution of Fats and Oils. Fats and fatty oils consist chiefly of glyceryl triesters (commonly known as glycerides) of saturated acids belonging to the fatty series and of related, unsaturated acids. Glycerol, the anhydride of which is the essential and characteristic constituent of all fatty molecules, is a trihydric alcohol. The acids combined with glycerol in edible fats and oils belong in three series as follows: (1) saturated acids  $(C_nH_{2n-2}O_2)$ , (2) unsaturated acids with one double bond  $(C_nH_{2n-2}O_2)$ , and (3) unsaturated acids with two double bonds  $(C_nH_{2n-4}O_2)$ . By far the most important of these acids are stearic acid  $(C_{18}H_{36}O_2)$  and palmitic acid  $(C_{16}H_{32}O_2)$ , belonging to the first series, and oleic acid  $(C_{18}H_{34}O_2)$ , belonging to the second series. The table on page 140 includes these and other acids occurring in edible fats and oils.

Acids. The difference in the molecules of stearic acid, belonging to the saturated or fatty series, and oleic acid with the same number of carbon atoms, belonging to the unsaturated series with one double bond, is brought out by the following structural formulas:

The double bond in the middle of the oleic acid chain should be noted. Because of this structure oleic acid, like other unsaturated acids, is more readily oxidized than the corresponding saturated acid, and forms halogen compounds analogous to the saturated molecule of stearic acid, thus explaining the theory of the iodine number determination (p. 152).

ACIDS OF FATS AND OILS USED AS FOODS (LEWKOWITSCH1)

Acid.	Formula.	Occurrence.			
Saturated Acids C <sub>n</sub> H <sub>2n</sub> O					
Butyric	$C_4H_8O_2$	Butter			
Caproic	$C_6H_{12}O_2$	Butter, cocoanut, palm nut			
Caprylic	$C_8H_{16}O_2$	Butter, cocoanut, palm nut			
Capric	$C_{10}H_{20}O_{2}$	Butter, cocoanut, palm nut			
Lauric	C12H24O2	Cocoanut, palm nut			
Myristic	$C_{14}H_{28}O_{2}$	Cocoanut, sesame, palm nut, butter			
Palmitic	C16H32O2	Most fats and oils			
Stearic	C18H36O2	Fats and most oils			
Arachidic	$C_{20}H_{40}O_{2}$	Peanut, butter <sup>2</sup> , rape, cocoa . cov			
Behenic	C22H44O2	Rape, mustard			
Lignoceric	$C_{24}H_{48}O_{2}$	Peanut			
Unsaturated Acids	1	·			
$C_nH_{2n-2}O$					
Hypogæic	$C_{16}H_{30}O_{2}$	Peanut			
Oleic	$C_{18}H_{34}O_{2}$	Most fats and oils			
Iso-oleic	$C_{18}H_{34}O_{2}$				
Rapic	$C_{18}H_{34}O_{2}$	Rape, mustard			
Erucic	C22H42O2	Rape, mustard			
$C_nH_{2n-4}O$		• ′			
Linolic	C <sub>18</sub> H <sub>32</sub> O <sub>2</sub>	Linseed, olive, cotton seed, peanut, sesame, cocoa, poppy seed, sunflower.			

<sup>1</sup> Chemical Technology and Analysis of Oils, Fats and Waxes. 5th Ed.

Saponification. Glycerides saponified by heating with concentrated sulphuric acid or slaked lime yield glycerol and the organic acids by catalysis. When the saponification is

<sup>&</sup>lt;sup>2</sup>Trace

effected by boiling with sodium or potassium hydroxide, alkali salts of the organic acids (soaps) and glycerol are obtained. In the determination of saponification number (p. 155), which is the number of milligrams of potassium hydroxide required to saponify one gram of fat, the result obtained is inversely proportional to the average molecular weight of the glycerides present.

Solubility and Volatility of the Acids. Butyric acid is soluble in water and distills without decomposition with steam. As the number of atoms in the acid molecule increases the solubility and volatility decrease, the former, however, more rapidly than the latter. The methods of determining soluble acids and soluble and insoluble volatile fatty acids (Reichert-Meissl and Polenske numbers) in fats and oils are based on these properties of the component acids.

Chemical and Physical Constants. In examining fats and oils with reference to their identity and purity complete analyses, involving determinations of the individual acids even if possible, would involve a vast amount of labor and be difficult to interpret. Instead of such analyses, determinations are made of the socalled "constants," dependent on the chemical and physical properties of the glycerides, or of the acids combined as glycerides. Owing to the somewhat variable amounts of the different glycerides in a given commercial fat or oil the constants vary within certain limits and are not, therefore, such definite figures, as for example, the melting- or boiling-point of a single pure glyceride. Allowance must always be made for this variation in deciding as to the identity or purity of a given fat or oil and especially in calculating the proportion of the constituents in a mixed fat from the results obtained in the determination of the constants. The following are the most important constants:

Physical Constants: Specific gravity, refractive index, melting-point, solidifying-point, viscosity, rise of temperature with sulphuric acid (Maumené test), heat of bromination.

Chemical Constants: Iodine number (Hübl or Hanus number), saponification number (Koettstorfer number), soluble

volatile fatty acids (Reichert-Meissl number), insoluble volatile fatty acids (Polenske number), soluble fatty acids, insoluble fatty acids, bromine number, acetyl value, free fatty acids, unsaponifiable matter.

All of the above constants are determined directly on the fat or oil; some of them, notably the solidifying-point (titer test), melting-point, and the iodine number, are also applicable to the mixed fatty acids obtained by saponification.

The table on p. 143 gives the range in specific gravity, refractive index, iodine number, saponification number, and volatile fatty acids of the most important edible fats and oils. Methods of determining the five constants selected, which are those most commonly determined, are described in the following sections. No figures are given for oleo oil, beef or cotton seed stearin, or the various mixtures used as butter and lard substitutes, as these are more or less indefinite in composition.

\*Material for Laboratory Practice. Most of the materials given in the table on p. 143 may be obtained of the grocer, the butcher, or the druggist; the remainder from dealers in laboratory supplies. Butter fat is obtained from butter by melting and decanting off the clear fat onto a filter. A sample of oleomargarine fat may be obtained in the same manner. Beef tallow may be rendered in the laboratory from suet; mutton tallow being of nearly the same composition may be dispensed with. So-called "compound lard," a lard substitute consisting of a mixture of cotton seed oil with enough beef or cotton seed stearin to harden it and a certain amount of real lard to impart flavor, should also be included.

The source and method of manufacture of the different fats and oils need not here be considered. Most of them are well-known products in the United States; sesame and rape oil, however, are less often met with. Sesame seed is extensively grown in India and other Oriental countries, where both the oil and the cake are important foods. Rape oil is obtained from the seeds of various species of the mustard family grown in Europe and the East. Cocoa butter, the fat expressed from cocoa mass

or chocolate in the manufacture of cocoa, is hard and waxy, cocoanut oil, on the other hand, a very soft fat at ordinary temperatures and a liquid on hot days, is classed, as the name implies, with the oils.

CONSTANTS OF EDIBLE FATS AND OILS (As given by Lewkowitsch and others)

	Specific Gravity at 15.5° C.	Refractive Index.	Iodine Number.	Saponifica- tion Number.	Volatile Fatty Acids (Reichert- Meissl Number).
Olive oil	0.916918	1.4660-1.4680 <sup>1</sup>	79–88 ³	185–196	0.6
Cottonseed oil	.922925	I.4700-I.4725 1	104-110	193-195	1.0
Peanut oil	.917921	1.4690-1.4707 <sup>1</sup>	83-103	190-196	
Sesame oil	.923924	1.4704-1.47171	103-115	189-193	1.2
Rape oil	.913917	I . 4710 <sup>1</sup>	94-105	170-179	0.6
Cocoanut oil	.912	1.44812	8-9.5	246-268	7-8.4
Butter fat	.926940	1.4590-1.4620 <sup>1</sup>	26-38	227	25-30.4
Beef tallow	.943952	1.4586 <sup>2</sup>	35-47	192-200	0.5
Mutton tallow	.937953	1.4586 <sup>2</sup>	32-46	192-195	
Lard	.934938	1.4584-1.46012	47-70	195	1.1
Cocoa butter	.950976	1.4566-1.4578°	32-41	192-202	0.2-0 8

<sup>&</sup>lt;sup>1</sup> At 25° C. <sup>2</sup> At 40° C.

So far as possible each student should be assigned a different fat or oil for determinations of specific gravity, refractive index, iodine number, and saponification number, the last two being determined in duplicate. Each student should also make a single determination of volatile fatty acids on both butter fat and oleomargarine fat, also apply the Halphen test for cotton seed oil and the Baudouin test for sesame oil, both to the oils themselves and to pure olive oil and in the case of Halphen test to pure and compound lard. One laboratory period is sufficient for these qualitative tests and the determination of the two physical constants, and one day for the determination of each of the three chemical constants, assuming that the reagents and standard solutions are furnished ready for use.

<sup>\*</sup> European olive oil; California olive oil sometimes runs over 90.

The chemical student doubtless will have already prepared and standardized sodium thiosulphate solution and tenthnormal sodium or potassium hydroxide solution, in which case these solutions may be used for the determinations of the iodine number and volatile fatty acids. The standardizing of half-

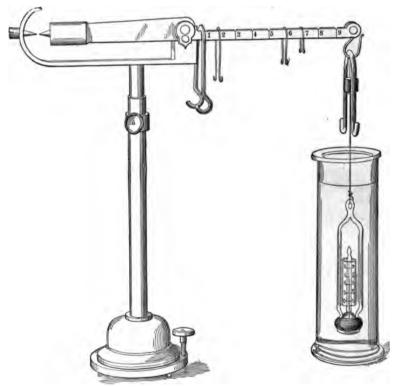


Fig. 88.—Westphal Balance.

normal hydrochloric acid, required for the determination of the saponification number, is described on p. 70.

\*Determination of the Specific Gravity with the Westphal Balance. The construction and method of use of the balance are evident from Fig. 88. Instead of weights there are four sizes of riders corresponding, when hung at the end of the beam,

to specific gravity readings of 1, 0.1, 0.01, and 0.001, respectively, and, when hung in the notches, to decimals of these readings as indicated by the numerals. In case two or more riders belong on the same notch the smaller should be suspended from one of the hooks of the larger. The thumbscrew at the base serves to bring the beam in exact equilibrium, as indicated by the points at the left, when the dry plummet is hung in the air. Distilled water at 15.5° C., by the thermometer in the plummet, should show a specific gravity of exactly 1.000. The specific gravity of the liquid shown in the cut is 1.1267, two of the largest size riders being used, one at the end and the other at 1 on the beam.

The specific gravity of the oils (except cocoanut) should be taken within a few degrees of 15.5° C. and the readings calculated to that temperature by the formula:

$$G = G' + 0.00064(T - 15.5),$$

in which G is the specific gravity at  $15.5^{\circ}$ , G' the specific gravity at  $T^{\circ}$ . Although the factor varies somewhat for the different oils the average (0.00064) is sufficiently accurate if the temperature does not vary greatly from  $15.5^{\circ}$ .

The fats, also cocoanut oil which at ordinary temperatures is a soft fat, should first be melted in a tall beaker over a piece of asbestos, using a low flame. Keeping the temperature as near constant as possible and only slightly above the melting-point, determine the specific gravity and note at the same time the exact temperature. Care must be taken that the melted fat is kept thoroughly mixed and consequently of uniform temperature throughout during the observation. As the melting temperature of hard fats must vary considerably from 15.5°, the factor peculiar to each fat must be used instead of 0.00064 in calculating to the standard temperature as follows:

FACTORS FOR CORRECTING SPECIFIC GRAVITY	(ALLEN)
Butter fat	0.000617
Tallow	.000675
Lard	. 000650
Cocoa butter	.000717

The specific gravity may also be determined in a pycnometer or by accurate spindles, but for practical work the Westphal balance is to be preferred. The temperature of boiling water, using a special heating apparatus, is often employed and the observed reading compared with the limits obtained on samples of known purity at that temperature.

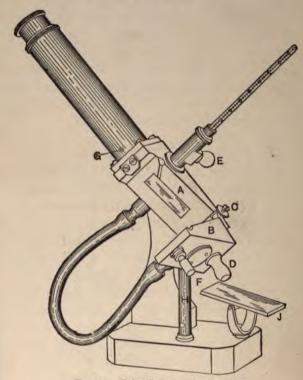


Fig. 89.—Zeiss Butyro-refractometer.

Determination of the Refractive Index. Two forms of tometer both made by Carl Zeiss, Jena, are suitable for camination of fats and oils. One of these, the butyroometer (Fig. 89), has an arbitrary scale on which the of refraction is observed directly through the eyepiece, ter, the Abbé refractometer (Fig. 90), gives readings in

terms of refractive index on the sector S after rotating the telescope F until the border line of total reflection passes through the point of intersection of two crossed lines. Both employ two Abbé prisms, A and B, on the lower one of which, when open (Fig. 89), a drop of the fat or oil is placed forming, when closed, (Fig. 90), a thin film through which the light passes after being

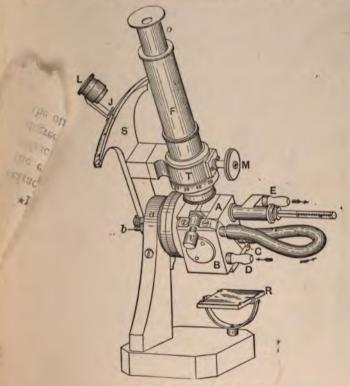


Fig. 90.—Abbé Refractometer.

reflected upward from the mirror. Both also are arranged to permit the passage of a stream of water of constant temperature through the two prisms, the temperature being indicated by a delicate thermometer.

The Abbé instrument, notwithstanding its more complicated construction and more cumbersome scale, is to be preferred for

general work, as it has a wider range, thus permitting its use for the examination of essential oils and the determination of the total solids of molasses, honey, and syrups from the refractive index. The scale reading obtained with one form of refractometer may be converted into the equivalent of the scale of the other form by calculation or reference to the table on p. 222.



Fig. 91.—Zeiss Immersion Refractometer.

A third form, known as the immersion refractometer (Fig. 91), used in the examination of milk serum, in the detection of methyl alcohol in liquors, etc., has a prism P, at the end of the tube which, for ordinary use, is plunged in a beaker containing the liquid under examination kept at a constant temperature in a glass-bottomed tank. At the left in Fig. 91, is shown a sec-

tion of the tube with a special metal beaker at the bottom for the examination of liquids excluded from the air.

\*Manipulation of the Abbé Instrument. Place the instrument in front of a window but not in the direct sunlight. Provide a large tank elevated about 2 ft. above the desk with a suitable arrangement for heating the water and a cock or siphon connected with a rubber tube for conducting a slow stream of the warm water to the refractometer. The tank should be large enough to hold water sufficient for the group of students using it on the same day. Heat the water to 40° C. for the examination of the more solid fats. Afterward water can be added and the temperature lowered to 25° C. for taking the readings on butter fat and the oils. The stream of water enters the lower prism (Fig. 90) at C, passes into the upper prism through the rubber tube and out of the latter at E. When the temperature, as shown by the thermometer in the upper prism, becomes constant release the lower prism by opening the screwhead v and allow it to swing into the position shown in Fig. 80. Smear a drop of the oil or melted fat on the glass surface, close, and again fasten in position with v. Rotate the tube with attached sector on the alidade until the border line appears in the field, then by means of the screwhead M, so adjust the compensator that the band of colors, due to dispersion, disappears and a sharp line of demarkation is obtained. Next rotate the tube until this line passes exactly through the intersection of the crossed lines of the instrument, making sure the temperature has become constant. Finally read the refractive index through lens L and record both this and the temperature.

If the temperature varies from  $40^{\circ}$  or  $25^{\circ}$  (T), as the case may be, correct the reading R' at T' to a reading of R by the formula R = R' - 0.000365 (T - T').

Leach and Lythgoe have devised a slide rule (Fig. 92), which not only converts the reading at one temperature to the reading at another, but also shows the butyro-refractometer readings corresponding to different refractive indices.

The refractometer furnishes the simplest means of dis-

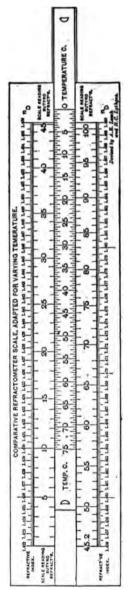


Fig. 92.—Leach and Lythgoe Slide Rule for Refractometer Calculations.

tinguishing olive oil, butter, and lard from imitations. The limits given in the table on p. 143 serve to distinguish olive oil from cotton seed and sesame oil. Wolny finds that the refractive index of the fat of pure butter at 25° C. ranges from 1.4590 to 1.4620 and of oleomargarine containing no real butter from 1.4650 to 1.4700. Lard, as given in the table on p. 143, has a refractive index at 40° C. of 1.4584 to 1.4601, while compound lard as found by the author shows a range of from 1.4606 to 1.4639.

\*Detection of Cotton Seed Oil by the Halphen Test. Olive oil was formerly extensively adulterated with cotton seed oil. Compound lard and other lard substitutes commonly consist largely of cotton seed oil and oleomargarine often contains cotton seed oil in addition to other fats.

The Halphen test <sup>1</sup> is carried out as follows: Place in a test-tube about 5 cc. of the oil or melted fat and the same volume of Halphen reagent, consisting of a mixture of equal volumes of carbon bisulphide, containing in solution 1 per cent of sulphur, and amyl alcohol. Mix and heat in a bath of boiling saturated brine for fifteen minutes. If cotton seed oil is present a deep red color is formed.

The constituent of the oil that gives the color, the identity of which has

<sup>&</sup>lt;sup>1</sup> Analyst, 1897, p. 326.

not yet been established, is destroyed or driven off by heating at 250 to 270° C. (Holde and Pelgry, Fulmer), but oil thus treated is not likely to be used. The amyl alcohol is useful because it contains pyridin (Gastaldi).

Another qualitative test is that first proposed by Bechi. It depends on the reduction of silver on heating the oil with a solution of silver nitrate in a mixture of alcohol and ether acidulated with nitric acid. This test is not entirely satisfactory, as rancid and overheated oils and fats not containing cotton seed oil often give a slight reduction. Milliau obviates this difficulty by applying the test to the fatty acids separated by saponification.

\*Detection of Sesame Oil by the Baudouin Test. Sesame oil is used as a substitute for and adulterant of olive oil. The addition of a small amount of sesame oil to oleomargarine is required by the laws of certain European countries so that the food inspector will readily be able to distinguish the product from butter by means of a simple qualitative test.

Apply the Baudouin test 1 to samples of olive and sesame oil as follows:

Dissolve 0.1 gram of cane sugar in 10 cc. of concentrated hydrochloric acid in a test-tube, add 20 cc. of the oil to be tested, and shake thoroughly for one minute. Allow to stand until the oil rises. If sesame oil is present to the amount of 1 per cent, the aqueous solution will become deep red.

Certain pure African olive oils are said to give a color, but of a different shade from that obtained with sesame oil. In doubtful cases the test may be applied to the fatty acids.

The test depends on the reaction of a minor constituent of the oil with the furfural formed by the action of the acid on the sugar. Villavecchia and Fabris<sup>2</sup> employ an alcoholic solution of furfural instead of sugar.

Other Qualitative Tests. Tolman's modification of Renard's test for peanut oil,<sup>3</sup> based on the separation of arachidic acid, is

<sup>&</sup>lt;sup>1</sup> Ztschr. angew. Chem., 1892, p. 509.

<sup>&</sup>lt;sup>2</sup> Jour. Soc. Chem. Ind., 1894, p. 13.

<sup>&</sup>lt;sup>3</sup> U. S. Dept. Agr., Bur. Chem., Bul. 77.

useful in the examination of olive oil. Palm oil used as a coloring for oleomargarine is detected by tests devised by Crampton and Simon.<sup>1</sup> Various tests are in use for detecting artificial colors, such as sulphur, annatto, carotin, and oil-soluble coal-tar dyes, in butter. Beef stearin, a common ingredient of lard substitutes, is detected by modifications of Belfield's microscopic test.

\*Determination of Iodine Number by the Hanus Modification of the Hübl Method. The formation of halogen addition compounds of the glycerides of the unsaturated acids occurring in fats has already been noted (p. 140). The determination of the iodine number serves to measure the degree of unsaturation. Two factors, involving the constitution of the fat, influence the results, (1) the nature of the unsaturated acids present, those with two double bonds absorbing a greater percentage than those with the same number of carbon atoms but with only one double bond, and (2) the molecular weight of the glycerides, those with low molecular weights absorbing a greater percentage than those of the same degree of saturation with high molecular weights. Of these factors the former is by far the most important. For example linseed oil has a very high iodine number (about 175), due to the presence of a considerable amount of linolic acid with two double bonds, while cocoanut oil, which consists largely of saturated acids, has a very low number (less than 10).

The original method of determining iodine number devised by Hübl, which for many years was exclusively used, employed a solution of iodine and mercuric chloride in 95 per cent alcohol. This solution deteriorated so rapidly in strength that after a few days it was useless, furthermore it acted so slowly on the fat that three hours' standing was required for the absorption of the iodine. Both of these defects are obviated by the solutions proposed by Hanus<sup>2</sup> and by Wijs, the former now being more generally used in the United States and the latter in England. The Wijs solution consists of iodine chloride dissolved in glacial

<sup>&</sup>lt;sup>1</sup> Jour. Amer. Chem. Soc., 1905, 27, p. 270.

<sup>&</sup>lt;sup>2</sup> Ztschr. anal. Chem., 1901, 4, p. 913.

acetic acid. Hanus in his modification of the Wijs solution employs iodine bromide.

Reagents: (1) Iodine Solution. Dissolve 13.2 grams of pure iodine in 1 liter of pure 99 per cent acetic acid and when the solution has cooled add 3 cc. of bromine. After the addition of the bromine the halogen content, as determined by titration against thiosulphate solution, should be nearly but not quite doubled.

(2) Decinormal Sodium Thiosulphate Solution. Dissolve exactly 24.8 grams of the c. p. crystallized salt in water and make up to 1 liter in a graduated flask. Unless the salt is impure or moist, which has never happened in the author's experience, the solution will be of the proper strength and further standardizing will be merely confirmatory.

The solution may be standardized by iodine, by potassium iodate, or by potassium bichromate. The iodine method, which is the oldest and in the author's experience the most accurate, is as follows: Tare a short glass tube, such as is used for weighing out the fat (Fig. 93), together with a microscopic cover glass; place in the tube about 0.2 gram of c. p. resublimed iodine, heat cautiously until the iodine melts, close with the cover glass, cool in a desiccator, and weigh. Dissolve the iodine in 15 cc. of 10 per cent potassium iodide solution, dilute with water, and add thiosulphate solution from a burette with stirring until only a yellow color remains, then add a little starch paste and continue the addition until the blue color is discharged. One cc. of the iodine solution should be equivalent to 0.0127 gram of iodine.

- (3) Starch Paste. Mix 1 gram of starch with 200 cc. of water, boil for ten minutes, and cool.
- (4) Potassium Iodide Solution. Dissolve 100 grams of the salt in water and make up to 1 liter.

Manipulation. Weigh a flat-bottomed glass cylinder about 10 mm. in diameter and 15 mm. high (Fig. 93). Transfer to the cylinder by means of a glass tube from 0.15 to 1.0 gram of the oil or melted fat, the quantity used being such as to absorb not more than 40 per cent of the iodine present in 30 cc. of the

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iodine solution. Use about (but no more than) 0.15 gram of olive, cotton seed, peanut, sesame, or rape oil, 0.25 gram of lard, 0.3 gram of beef or mutton tallow, 0.4 gram of butter fat or cocoa butter, and 1.6 gram of cocoanut oil. Weigh the cylinder



Fig. 93.—Iodine Number Apparatus. Introducing cylinder with fat into bottle. Cylinder natural size at right.

containing the oil or fat, in the latter case after cooling to room temperature. Because of the small quantities employed the weighing of the cylinder, both before and after adding the material, should be performed with the highest degree of accuracy which is possible because of the small size of the cylinders and the non-hygroscopic character of the fatty materials. No desiccator need be used.

By means of a pair of forceps carefully introduce the cylinder and contents into a glass-stoppered bottle of about 300-cc. capacity, add 10 cc. of chloroform and, after complete solution is effected,

introduce 30 cc. of the iodine solution with great care by means of a pipette. Shake gently and allow to stand in a dark place with occasional shaking for thirty minutes.

Add 15 cc. of potassium iodide solution and 100 cc. of water, then titrate slowly with standard thiosulphate solution, depending for an indicator first on the yellow color of the liquid and finally, when that has nearly disappeared, on the blue color obtained by adding a little starch paste. When the titration is nearly finished, stopper the bottle after each addition of thiosulphate, and shake to remove the iodine from the chloro-

form. In addition to duplicate analyses two blank determinations should be performed in exactly the same manner, using only the reagents.

In calculating the results subtract from the average number of cubic centimeters of thiosulphate solution, obtained in closely agreeing blank determinations, the number of cubic centimeters obtained in each actual analysis, and multiply the difference by 0.0127, thus obtaining the grams of iodine absorbed. To obtain the percentage of iodine absorbed, which is the iodine number, multiply the grams of iodine absorbed by 100 and divide by the weight of material employed.

\*Determination of the Saponification Number by the Koetts-torfer Method. This number <sup>1</sup> is a measure of the average molecular weight of the mixed glycerides constituting a given fat or oil. Although the range is not nearly so great as that of the iodine number, the saponification number is an important constant, particularly in distinguishing rape, mustard, and other cruciferous oils from most of the other edible oils, in identifying cocoanut oil, and in an exhaustive examination of butter suspected of sophistication.

Reagents. (1) Alcoholic Potassium Hydroxide Solution. Dissolve 30 grams of c. p. potassium hydroxide in 1 liter of 95 per cent alcohol, previously purified by standing some days with potassium hydroxide and distillation. The solution is approximately half-normal.

(2) Standard Half-Normal Hydrochloric Acid Solution.— Prepare in the usual manner and standardize as described on p. 70.

Process. Weigh accurately an Erlenmeyer flask of about 200-cc. capacity, introduce 2 to 2.5 grams of the oil or melted fat (1.5 to 2 grams of butter or cocoanut fat), and weigh again. Add 25 cc. of the alcoholic potassium hydroxide solution, connect with a reflux condenser, and boil gently, by heating over a piece of asbestos paper, for thirty minutes (Fig. 94). Cool, add a few drops of phenolphthalein solution as an indicator, and

<sup>&</sup>lt;sup>1</sup> Ztschr. anal. Chem., 1879, p. 199.

titrate the excess of alkali with standard half-normal acid solution.

Conduct two actual analyses, then two blanks in exactly

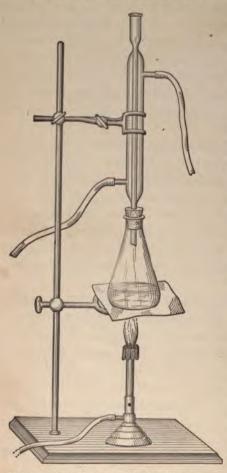


Fig. 94.—Saponification Number Apparatus.

the same manner. It is immaterial whether the quantity of potash solution discharged from the pipette is exactly 25 cc., but of great importance that the quantity is the same in all cases. Care must, therefore, be taken to use the same pipette and allow it always to drain for exactly the same length of time.

In calculating the results subtract from the average number of cubic centimeters of half-normal acid, obtained in closely agreeing duplicates, the number obtained in each actual analysis, multiply the result by 28.06 (the number of milligrams of KOH corresponding to each cubic centimeter of halfnormal acid), and divide the product by the weight of material em-

ployed. The Koettstorfer or saponification number thus calculated represents the number of milligrams of potassium hydroxide necessary to saponify completely I gram of the material. \*Determination of the Volatile Fatty Acids by the Leffmann and Beam Modification 1 of the Reichert-Meissl Method. This process is used chiefly in distinguishing oleomargarine from butter. It depends on the presence in butter fat of a considerable amount of glycerides of the fatty series with low carbon content, notably butyric acid (C<sub>4</sub>H<sub>8</sub>O<sub>2</sub>), whereas the fats and oils used in butter substitutes, known collectively as oleomargarine, contain only small amounts if any. These acids are volatile on distillation with steam and are also quite soluble in water. Reichert was the first to make use of these facts in a method for detecting oleomargarine. The process was later improved by Meissl and later still by Leffmann and Beam, the latter employing for saponification a mixture of glycerol and sodium hydroxide instead of alcoholic sodium hydroxide solution.

Polenske,<sup>2</sup> in 1904, further modified the process so as to determine the volatile acids insoluble, as well as those soluble, in water, thus differentiating cocoanut oil, which contains a considerable amount of glycerides of these insoluble volatile acids, from butter. The following figures illustrate the value of both determinations:

	Reichert-Meissl Number.	Polenske Number.
Butter fat, 31 samples, (Polenske)	23.3-30.I	1.5- 3.0
Cocoanut oil, 4 samples (Polenske)	6.8- 7.7	16.8-17.8
Oleomargarine fat (Arnold)	0.5	0.53
Lard (Arnold)	0.35	0.5
Tallow (Arnold)	0.55	0.56

By referring to the table on p. 140, it will be noted that butter contains acids with four to fourteen atoms of carbon and cocoanut oil, acids with six to fourteen atoms, both inclusive. It should be further noted that butter fat contains a considerable amount of the acid with four atoms of carbon (butyric),

<sup>&</sup>lt;sup>1</sup> Analyst, 1891, p. 153.

<sup>&</sup>lt;sup>2</sup> Arbeit a. d. Kaiserl. Gesundheitsamte, 1904, p. 545.

which is not found in cocoanut oil, while cocoanut oil contains considerable amounts of acids with twelve and fourteen atoms (lauric and myristic) which occur only in small quantities in butter fat. Since the solubility decreases as the number of carbon atoms increases, it is obvious why butter fat gives a

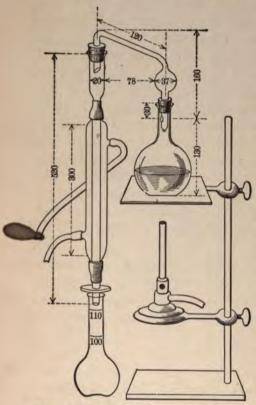


Fig. 95.—Distillation Apparatus for Volatile Fatty Acids.

high Reichert-Meissl number and cocoanut oil a high Polenske number

As cocoanut oil is not so commonly used in American oleomargarine as that made in Europe, only the soluble volatile acids or the Reichert-Meissl number need be determined by the student. Single determinations should be made on both butter fat and oleomargarine fat prepared as described on p. 142.

Method. Weigh accurately a 300-cc. Jena flask, introduce as much of the melted fat as will be delivered by a clean, dry 5-cc. pipette, and enough more to bring the weight up to about 5 grams. Allow to cool and weigh accurately flask and

fat. Add 20 cc. of glycerine and 2 cc. of a solution prepared by dissolving 100 grams of c. p. sodium hydroxide, free from carbonates, in 100 cc. of boiled water. Heat cautiously on a piece of asbestos paper until the fat is saponified, which requires about five minutes and is indicated by the clearing up of the boiling

liquid. While still hot add very cautiously, at first, drop by drop, to prevent foaming, 90 cc. of boiled water and shake until the soap is dissolved. The solution should be perfectly clear and nearly colorless. Rancid or oxidized fats that yield a brown soap should not be examined.

To the soap solution add 50 cc. of dilute sulphuric acid (25 cc. to 1 liter), and about 0.5 gram of granulated pumice stone with grains 1 mm. in diameter, then connect with a condenser, such as shown in Fig. 95, and distill at a rate sufficient to give a distillate of 110 cc. in about twenty minutes, using a stream of water that will cool the condensed liquid to about 20° to 30°. Cool in water of about 15°, make up to the mark, mix by inverting the flask four or five times, filter through an 8-cm. dry filter, and pipette 100 cc. of the filtrate into a beaker. Titrate with tenth-normal alkali, using a few drops of phenolphthalein solution as an indicator.

If exactly 5 grams of fat were used the number of cubic centimeters of standard alkali required multiplied by 1.1 is the Reichert-Meissl number, otherwise calculate to that amount.

Determination of the Polenske Number. In this determination (which for reasons already stated may be omitted), the condenser tube, flask, and filter, after obtaining the Reichert-Meissl number, as described in the preceding section, are washed with three 15-cc. portions of water and the insoluble volatile acids dissolved by the same treatment, using 15-cc. portions of neutral 90 per cent alcohol. The united alcoholic washings are finally titrated as in the determinations of the soluble acids.

Other Constants of Fats and Oils. The Melting-point of Fats<sup>1</sup> is determined in a capillary tube similar to that used for crystalline substances except that it is open at both ends, the melted fat being drawn up into the tube and allowed to solidify (Fig. 96). After twelve hours' cooling the tube is attached by a rubber band to the bulb of a delicate thermometer and both are suspended in a test-tube of water supported in a flask also containing water (Fig. 96).

<sup>&</sup>lt;sup>1</sup> Leach's Food Inspection and Analysis, p. 480.

The flask is gradually heated until the fat melts.

The Maumené Test 1 is the measure of the rise of heat with sulphuric acid, which is highest with oils containing the greater

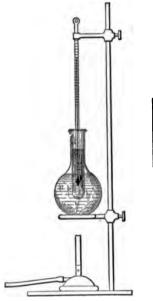


Fig. 96.—Melting-point Apparatus for Fats.

percentages of unsaturated acids and, therefore, having the highest iodine numbers.

The Bromination Test is similar to the last in principle, bromine being used instead of sulphuric acid.

Another physical test is the solidifying point of the fatty acids, known as the *Titer Test*, determined by the Dalican method.

Most of the tests designed primarily for the oils or fats themselves may also be determined on the fatty acids liberated by a mineral acid after saponification. Naturally the results are not the same as those obtained by the direct determination.

Among the chemical tests is the estimation of the Soluble and In-

soluble Fatty Acids. In the method for the determination of the Reichert-Meissl number the separation of the insoluble acids as an oily liquid, after addition of sulphuric acid to the saponified fat, is evident. The method for determining the soluble and insoluble acids also involves saponification and separation of the acids with a mineral acid. The soluble acids are determined by titration; the insoluble acids by direct weighing of the washed and cooled oily layer. The method is now of comparatively small importance.

The method for estimating the *Acetyl Value*, first proposed by Benedict and later modified by Lewkowitsch, depends on the substitution of the hydrogen of the alcoholic hydroxyl group

<sup>&</sup>lt;sup>1</sup> Comptes rendus, 1852, 35, p. 572.

by the acetic acid radicle on heating with acetic anhydride, the acetylated fat being subsequently saponified and the acetic acid separated and titrated.

Fats and oils contain, in addition to glycerides, very small amounts of *Unsaponifiable Matter* such as *Cholesterol* and *Sitosterol*. The former is found in animal, the latter in vegetable fats.

Hydrogenation of Oils. The hardening of the oils by hydrogenation, using nickel or some other metallic catalyzer, is now practiced on a commercial scale, cotton seed and other vegetable oils being thus changed into hard fats by the conversion of the olein into a saturated glyceride such as stearin. This treatment takes the place of adding to oils a hard fat such as stearin for the purpose of imitating the consistency of lard and other semisolid fats. It also adds to the list of edible oils, whale oil, which is not only hardened, but freed from rank-tasting impurities. This process changes materially the constants of the oil, thus increasing greatly the difficulties in interpreting the results of analyses.

## CHAPTER VIII

# FRUITS, FRUIT PRODUCTS, LIQUORS, AND VINEGAR

In ascertaining the food value of fruits and vegetables, as well as their products, the percentages of water, fat, fiber, protein, ash, and nitrogen-free extract, determined by practically the same methods as are used for grain, seeds, and their products, are of first importance.

Sugars. Determinations of sugars are of special value in the examination of fruits as sucrose and invert sugar, the former being largely transformed into the latter during ripening, are usually present, contributing to the immediate food value and furnishing the material for alcoholic fermentation of the fruit juices in the manufacture of wines and ciders and for the subsequent acetous fermentation in the manufacture of vinegar. Sweetened fruit products, such as preserves, jellies, and fruit syrups, contain sucrose and invert sugar as their chief constituents, which are determined by the same methods as are used in the analyses of sugar, molasses, and other cane and beet products.

Acids. As all succulent fruits contain one or more organic acids, such as malic, citric, and tartaric, which, in the case of vinegars, are supplemented by acetic acid, no analysis of a fruit product is complete that does not include the amount of acidity. Estimating this acidity by titration with a standard alkali solution does not differentiate between the different acids, although, in many cases, the chemist knows the acid present in a given product to the practical exclusion of all others, thus permitting the use of the proper factor for the calculation of the percentage of that acid from the volume of standard alkali solution employed. For example, the acidity of vinegar would

be calculated as acetic, disregarding the small amount of malic acid in cider vinegar and of tartaric acid or cream of tartar in wine vinegar, and the acidity of lime juice would be calculated as citric acid.

Starch, Oil, and Fiber. A few fruits, notably the banana, are distinctly starchy, although at full maturity the starch passes largely into sugar, and one common fruit, the olive, is very oily. Most fruits, however, contain only small amounts of these constituents. Fruit juices are not only free from starch and oil, but also from crude fiber, therefore the analysis of the juices and of the liquors and vinegars made from them would not include determinations of these three constituents.

Alcohol and Other Constituents. The labor saved in omitting analyses for starch, oil, and fiber in liquid products is offset by the need of determining alcohol in alcoholic liquors and acidity in most fruit products as well as of a number of substances present in small amount in liquors and vinegars which, although of little or no food value, serve as indications of strength and purity. The calculation of the alcoholic strength of liquors from the specific gravity of the distillate is of great value in industrial work and in the enforcement of excise and adulteration laws.

Solids. While in dry products such as flour, meal, and cattle foods there is no ocular indication of the presence of moisture, in liquid fruit products there is no appearance of solid matter. We accordingly give the results in the former case in terms of moisture, in the latter in terms of total solids or extract. In juices the extract consists largely of sugar, which disappears almost entirely on fermentation.

\*Laboratory Practice. The purpose of the two laboratory exercises which follow is partly to aid the student in a practical understanding of a few important analytical processes and partly to show how these processes are applied in scientific and technical investigations, having in mind the formation of alcohol from sugars and of acetic acid from alcohol with the consequent disappearance of most of the solid matter.

Material for Practice. As representative fruit juices either sweet cider or grape juice may be selected, both products being obtainable bottled and sterilized. Fermented cider sampled after the escape of carbon dioxide has ceased or an unsweetened wine, such as claret, will serve as a suitable alcoholic beverage, and either cider or wine vinegar as an acetified liquid. The most interesting sets of samples are those made from the same lot of apple or grape juice, the sterilized juice, the cider or wine, and the vinegar being bottled at the suitable time. Lacking these the ordinary commercial products will answer. It is recommended that products of the same fruit, either apple or grape, be analyzed by the same student. In most sections of the United States the apple series will be most readily obtainable and the three products can be kept in bottles for years.

The student should carry on determinations of solids and acidity, also tests for sugar, in the three products of the series, on the first day. On the second day he can give his attention largely to the determination of alcohol. All the results should be calculated as grams per 100 cc., which is approximately the same as grams per 100 grams or true percentage by weight.

The average of several analyses of each of the three products follows:

AVERAGE COMPOSITION OF APPLE JUICE, FERMENTED CIDER, AND CIDER VINEGAR

	Number of Analy- ses Aver- aged.	Solids.	Total Sugar as Invert.	Malic Acid.	Acetic Acid.	Alcohol.	Ash.
Apple juice (Browne) Fermented cider	10	13.21	11.72	0.73		****	0.28
(Browne)	4	2.46	0.40	0.25	0.34	5.40	0.26
Cider Vinegar (Lythgoe)	22	2.49	0.25	0.11	4.84		0.34

# FRUIT JUICES

\*Determination of Total Solids. The determinations of chief importance are of solids, sugars, and acidity. As the solids consist largely of sugars, fruit juices may be regarded as dilute sugar syrups and the solids may be approximately estimated from the specific gravity or the refraction, using certain tables which have been prepared for the purpose. These methods have the advantage of rapidity, and the inaccuracy, due to the presence of solids other than sugar such as organic acids, is offset by the inaccuracies of a gravimetric determination due to the difficulty of removing all the water on the one hand and the decomposition of levulose during heating on the other. latter error is obviated by drying in vacuo at 70° C., but the process is tedious and requires special apparatus. For many purposes drying in an open dish at 100° for a conventional time is satisfactory, the decomposition not being sufficient to affect the general conclusions.

In this connection it may be stated that a method may not have the highest degree of scientific accuracy and yet be quite as useful for certain purposes as if it were absolutely exact. This is because it is often the relative rather than the absolute results that are desired, and also because by experience the analyst learns to interpret his analyses in terms of yield of alcohol or acetic acid by the commercial process.

Method. Weigh a flat-bottom tinned lead or aluminum dish such as is used in determining milk solids (p. 15), introduce 5 cc. of the juice measured from a pipette, evaporate on a water bath to dryness, making sure that the liquid is distributed over the bottom of the dish, and dry in a water oven at the temperature of boiling water for two and one-half hours. Cool in a desiccator and weigh. All this can be done in a single laboratory period. Calculate the weight of solids in 100 cc. of the juice. If drying dishes or oven capacity are insufficient for duplicate determinations, one will answer. The process is so simple that errors of manipulation are not probable, further-

more the results of the different students should check each other.

Carry along determinations on the fermented cider and vinegar by the same method and at the same time.

\*Determination of Sugar. The sugar in a fruit juice freshly expressed is usually a mixture of sucrose and invert sugar, the latter being formed from the former during ripening. Further change of the sucrose to invert sugar goes on in the juice during storage and is accelerated during sterilization, consequently the sugar in the samples of sweet cider and grape juice used for laboratory practice, especially if sterilized, may consist entirely of invert sugar. In order to be certain of complete inversion, treatment with acid is necessary preliminary to copper reduction, but for our purpose it will be sufficient to boil for two minutes 1 cc. of the fruit juice directly with 50 cc. of water and 25 cc. each of copper sulphate and alkaline Rochelle salts solutions (p. 76), noting that a copious precipitate of copper suboxide is formed.

Should the student have opportunity the quantitative determination may be carried out as follows:

Method. Pipette 5 cc. of the cider or grape juice into a 100-cc. graduated flask, dilute with about 50 cc. of water, and add lead subacetate solution (p. 133), drop by drop, until with shaking a precipitate no longer forms. Dilute to the mark, shake, and filter through a dry filter into an Erlenmeyer flask. To the filtrate add dry powdered potassium oxalate with shaking until all the lead is precipitated. Filter through a dry filter into a small Erlenmeyer flask. Pipette 50 cc. of the filtrate and 25 cc. of water into a graduated 100-cc. flask, add 5 cc. of concentrated hydrochloric acid, and invert in a water bath kept at 72° to 73°, exactly as described on p. 131. Cool, add sodium hydroxide solution until slightly alkaline to litmus paper, then add hydrochloric acid drop by drop until the paper turns red, make up to the mark, and shake. If the solution is not entirely clear, filter through a dry filter.

Determine the copper-reducing power of 50 cc. of the solu-

tion by the Munson and Walker method as described on p. 76, except that the weight of invert sugar, corresponding to the copper suboxide, should be found in the table (pp. 213 to 221), and the weight of this sugar in 100 cc. of the original cider or juice calculated. A comparison should be made of the results obtained for solids and sugars.

\*Determination of Acidity. Of the non-sugar solids, organic acids and ash are the chief constituents. Determine the acidity by titrating 25 cc. of the cider or juice with tenth-normal sodium hydroxide solution. For the cider use as indicator a few drops of phenolphthalein solution (1 gram in 100 cc. of alcohol), for the grape juice, so-called neutral litmus paper. Titrate also the fermented cider or wine and the vinegar at the same time, using for the vinegar only 10 cc.

## WINE, CIDER, AND OTHER LIQUORS

Fermentation. Grape must, cider, and other fruit juices ferment through the action on the invert sugar of the enzyme Zymase of the wild yeast plants Saccharomyces ellipsoideus, S. apiculatus, etc., which naturally occur on the outside of the fruit and find their way into the expressed fruit juices, the reaction being as follows:

$$\begin{array}{c} C_6H_{12}O_6 = 2C_2H_6O + 2CO_2 \\ \text{Dextrose or} \\ \text{levulose} \end{array}$$

In the manufacture of malt liquors the conversion of starch into the soluble carbohydrate *Maltose* is first effected by means of *Diastase*, the enzyme of malt, then the maltose is hydrolized by means of an enzyme in yeast, known as *Maltase* or maltoglucase, with the formation of dextrose as follows:

$$2C_{6}H_{10}O_{5} + H_{2}O = C_{12}H_{22}O_{11}$$
Starch Maltose
$$C_{12}H_{22}O_{11} + H_{2}O = 2C_{6}H_{12}O_{6}.$$

For the fermentation of malt liquors yeast of the species Saccharomyces cerevisiæ is added. In making lager beer a strain known as bottom yeast (Fig. 97) is used while for ale top yeast (Fig. 98) is necessary.

Liebig regarded fermentation as a purely chemical process

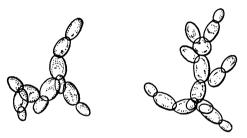


Fig. 97.—Bottom or Beer Yeast. Budding plants. (LINDNER.)

and ignored the biological theories of Pasteur and others which have since been accepted. Kröber by his classical researches has more recently shown that the ferments of yeast may act without the intervention of the growth of the cells,

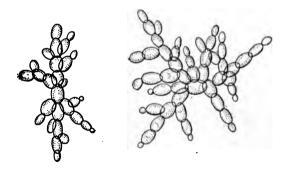


Fig. 98.—Top, Ale, or Distillery Yeast. Budding plants. (LINDNER.)

thus returning in a sense to the purely chemical theories of Liebig.

The carbon dioxide formed during fermentation is either allowed to escape or else, as in the case of malt liquors and

effervescent wines, is confined, at least in part, by tight casks or corked bottles.

Natural wines cannot contain more than 18 per cent of alcohol, as the yeast plant ceases to grow after that strength has been reached. By adding alcohol, fortified wines, such as sherry and port, are obtained and by distillation any desired alcoholic strength can be secured. Cognac or French brandy is the distillate from wine, cider brandy, from fermented cider, whiskey and gin, from fermented grain infusions, and rum, from diluted and fermented molasses.

Theoretically over 51 per cent of invert sugar is obtainable as alcohol, but practically under the most favorable conditions the yield is less than 49 per cent, the remainder going to form glycerol, succinic acid, and various higher alcohols which make up the fusel oil of distilled liquors.

Analysis of Liquors. In addition to alcohol, the characteristic constituent of all fermented and distilled liquors, the following minor constituents are determined:

Wines and Ciders. Extract or solids, sugars, acids (fixed and volatile), tartaric and malic acids (free and combined), glycerol, potassium sulphate (used in plastered wines), sodium chloride, nitrates, tannin, preservatives, and colors.

Malt Liquors. Extract, sugars, dextrin, glycerol, acids (fixed and volatile), protein, phosphoric acid, added bitter principles and preservatives, and arsenic (introduced in glucose made with impure acid).

Distilled Liquors. Extract, acids, esters, aldehydes, furfural, fusel oil, added wood alcohol, and caramel (added for coloring).

The complete analysis of a liquor is a laborious task, but such an analysis is not ordinarily necessary except in special cases as in detecting adulteration, in tracing the cause of certain defects, or as a guide in special manufacturing problems.

The Composition of the most important wines, malt liquors, and distilled liquors appears in the following tables:

# AVERAGE COMPOSITION OF EUROPEAN WINES (KOENIG) Results expressed as grams per 100 cc.

	Alcohol.	Extract.	Total Acidity as Tartaric.	Volatile Acids as Acetic.	Sugar.	Gly- cerol.	Ash.	Phos- phoric Acid.
Claret		2.42 2.91 3.03 4.06 2.36	0.58 0.77 0.66 0.41 0.61	0.10 0.05 0.09 	0.23 0.23 0.84 2.40 0.53	0.73 0.85 0.97 0.51 0.71	0.20 0.25	0.029 0.045 0.032 0.028

# AVERAGE COMPOSITION OF MALT LIQUORS (KOENIG)

	Alcohol by Weight.	Extract.	Acid as Lactic.	Gly- cerol.	Ash.	Phos- phoric Acid.	Nitro- genous Sub- stances.	Sugar as Maltose.
Lager beer	3-93	5.79	0.15	0.17	0.23	0.077	0.71	0.88
Bock beer	4.69	7.21	0.17	0.18	0.26	0.089	0.73	1.81
Ale	4.75	5.65	0.28		0.31	0.086	0.61	1.07
Porter	4.70	6.59	0.28	• • • •	0.36	0.093	0.65	2.62

## COMPOSITION OF DISTILLED LIQUORS

	( and a second	t by	GRAMS PER 100 LITERS OF PROOF SPIRITS.							
		per cent		Acids.						
	Analyst.	Alcohol, pe Volume.	Extract.	Total.	Volatile.	Esters.	Aldehyde.	Furfural.	Fusel Oil.	
Whiskey:						7				
Scotch, 8 yrs. old	Vasey	1		Carrier.	24.0	44.8	7.1	2.0	100.0	
Irish, 7 yrs. old	Vasey	9.9.8			20.9	10.5	5.6	1.7	102.0	
, Rye, 4 yrs. old	Crampton	55.6	185.0	65.9		69.3	13.9	2.8	125.1	
Bourbon, 4 yrs. old	Tolman	52.2	151.9	58.4	500	53.5	11.0	1.0	123.9	
Imitation rye	Ladd	45.0	506.I	10.6	7.3	17/20/20	trace	0.0	46.9	
Cognac, 10 yrs. old	Vasey				37.2	54.6	8.3	0.0	62.1	
Rum	Vasey	1	0000		14.0	199.5		1.4	45.3	
Gin	Vasey	***			0.0	18.7		0.0	22.3	
Neutral spirits	Ladd	94.0	1,2	3.8	3.8	14.0	100	trace	14.8	

<sup>&</sup>lt;sup>1</sup> Includes caramel color.

\*Determination of Alcohol. The method of determination is the same for all kinds of alcoholic liquors except that the addi-

tion of 0.1 to 0.12 gram of calcium carbonate or standard alkali to neutral reaction is necessary if the wine or cider has partly turned into vinegar and only 25 grams or cc. of distilled liquors and cordials are employed.

Method. The distillation apparatus used for determining the volatile fatty

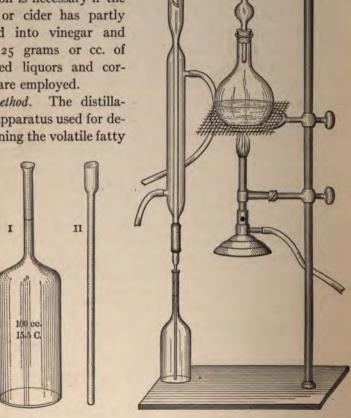


FIG. 99.

FIG. 100.

Fig. 99.—I Pycnometer; II Delivery Tube. Fig. 100.—Alcohol Distillation Apparatus.

acids (Fig. 95) is suitable except that a delicate pycnometer (Fig. 99, I), is substituted for the wide-mouthed receiving flask and the condenser tube is connected at the lower end by means

of a rubber tubing with a delivery tube (Fig. 99, II), the lower part of which is of such a size that it readily passes through the neck of the pycnometer. The height of the pycnometer should be such that it can stand erect on the balance pan and the inside of the neck should be 5 mm. It is calibrated to contain 100 grams of water at 15.5° C. Fig. 100 shows the complete apparatus for alcohol determination set up ready for use.

If the liquor is effervescent pour from one glass to another until no more bubbles of carbon dioxide escape. Weigh the clean, dry pycnometer, introduce the delivery tube, and attach the latter to the condenser tube. Pipette 100 cc. of the sample into a 300-cc. flask, add 50 cc. of water and a little tannic acid to prevent frothing. Attach to the condenser, turn on the water, heat cautiously to boiling, and continue to boil until the pycnometer is filled nearly to the bottom of the neck. Detach the delivery tube, rinse with a few drops of water, and mix by shaking. Add water nearly to the graduation mark and place in a bath of water at 15.5°, taking care that the water covers the pycnometer to the height of the liquid within. After standing in the bath at least fifteen minutes, remove the pycnometer, without delay add water at 15.5° by means of a small pipette until the lower meniscus is exactly at the mark, dry off the outside surface, and weigh.

Subtract from the total weight the weight of the empty pycnometer, thus obtaining the weight of the distillate, which divided by 100 gives its specific gravity. In the table on pp. 226 to 230 find the grams of alcohol per 100 cc. corresponding to the specific gravity, which is the common way of expressing the result in wine analysis, also the percentage of alcohol by volume and by weight in the distillate. As the volume of the sample and of the distillate are both 100 cc., the grams of alcohol per 100 cc. and the percentage by volume in both are the same. To obtain the percentage by weight multiply the weight of the distillate by the percentage of alcohol by weight contained in it and divide by the weight of the sample obtained either by a direct weighing of 100 cc. or from the specific gravity.

\*Determination of Solids (Extract). As is true of fruit juices the extract in sweet wines (sherry, port, etc.), cannot be determined with absolute accuracy by drying at 100°, owing to the decomposition of levulose. In the case of claret, Rhine wine, and others containing less than 3 per cent of extract, 50 cc. may be evaporated to dryness in a flat-bottomed dish 85 mm. in diameter and dried for two and one-half hours at 100° C., as prescribed by the German official method.

In the analysis of the fermented cider or light wines selected for laboratory practice satisfactory results may be obtained by evaporating 10 cc. of the wine or cider in a tinned lead or aluminum dish 65 mm. in diameter, such as is used for milk solids, and drying two and one-half hours at 100° C. This work is carried out in connection with the determination of solids in the juice and the vinegar.

\*Determination of Acidity. Total acidity is found by the same method as is used for sweet cider or grape juice and vinegar.

Volatile Acidity is valuable in wine analysis, as it is a measure of souring or incipient acetous fermentation. The process consists simply in distilling a portion of the wine and titrating the distillate. This need not be carried out by the student.

#### VINEGAR

Kinds of Vinegar. Any alcoholic liquor of suitable dilution may be subjected to acetous fermentation for the manufacture of vinegar. On the Continent Wine Vinegar is commonly made from white or red wine, the former being the better. In England Malt Vinegar is preferred, while in the United States Cider Vinegar is regarded as the standard product. Owing to the high price of cider vinegar and the increased demand, large quantities of Distilled Vinegar are now made from dilute alcohol, the process being carried on in conjunction with the manufacture of compressed yeast. While the distilled product is quite as strong as cider vinegar, it is lacking in ethers and other flavoring constituents and contains only a very small

amount of sugar, phosphates, and other solids, glycerol and other constituents characteristic of vinegar made from fermented liquors.

Sugar Vinegar or Molasses Vinegar and Glucose Vinegar are made in considerable quantities. Dilute acetic acid obtained by purifying pyroligneous acid from the dry distillation of wood is not regarded as suitable for food.

Analyses of different kinds of vinegar appear in the table on p. 176.

Process of Manufacture. Mycoderma aceti, the bacterium which converts the alcohol into acetic acid (Fig. 101), is widely

distributed and the spores are likely to find their way into the barrels of cider or other liquor stored with open bungholes in the farmer's cellar. The process is commonly accelerated by adding the slimy



Fig. 101.—Vinegar Bacteria, Mycoderma aceti. (Fischer.)

growth known as "mother of vinegar" from a barrel containing vinegar already made or in process of making.

Farmer's or barrel-fermented vinegar requires two or three years for developing its full acid strength, owing partly to unfar vorable temperatures, but chiefly to insufficient contact with the oxygen of the air, acetous fermentation, unlike alcoholic fermentation, being an oxidation process, as shown by the following equations:

Quick-process or generator vinegar is made by allowing the cider to drip through beech shavings, previously soaked in old vinegar, contained in a cask or vat through which passes a current of warm air. By carefully regulating the conditions the vinegar is formed in a few days.

Composition of Vinegar. The following table gives the average composition of cider, wine, malt, and distilled vinegar:

AVERAGE COMPOSITION OF DIFFERENT KINDS OF VINEGA	AVERAGE	COMPOSITION	OF	DIFFERENT	KINDS	OF	VINEGAR
--	---------	-------------	----	-----------	-------	----	---------

Kind of Vinegar.	Number Samples Ana- lyzed.	Acidity as Acetic Acid.	Organic Acid other than Acetic.	Total Solids.	Sugars.	Ash.	Phosphoric Acid (PrOs).
Cider (Lythgoe)	22	4.84	0.11 <sup>1</sup>	2.49	0.25	0.34	0.035
Wine (Koenig)	17	5 · 57	0.132	1.89	0.35	0.27	0.053
Malt (Hehner) Distilled (Paris Munici-		4.23	0.11 1	2.70		0.34	0.105
pal Lab.)		6.34		0.35	trace	0.04	

<sup>&</sup>lt;sup>1</sup> Malic, free and combined.

From the figures in the table it is evident that distilled vinegar is readily distinguished from cider, wine, and malt vinegars by the low percentages of total solids and ash. As a safeguard against adulteration with distilled vinegar, as well as dilution, the Federal standard and the laws in certain states require that cider vinegar contain at least 4 grams of acetic acid and 1.6 grams of solids in 100 cc. But a minimum figure for solids alone does not suffice, as boiled sweet cider can readily be added in sufficient amount to bring the percentage of solids above the limit. To prevent this fraud, the standard requires that the solids contain not more than 50 per cent of reducing sugars, and also fixes the minimum percentages of ash, phosphoric acid, and alkalinity of ash.

To illustrate, 1 part of sweet cider containing over 13 per cent of solids, the average of Browne's results (p. 165) mixed with 7 parts of distilled vinegar would contain over 1.60 per cent of solids, but the amount of sugar in the solids would greatly exceed 50 per cent; furthermore the minimum limits for ash, phosphoric acid, and alkalinity of ash would not be reached. Frear, who suggested the ratio of sugars to total solids as required in the above standard, also pointed out the importance of the

<sup>&</sup>lt;sup>2</sup> Tartaric, free and combined.

ratio of ash to total solids. Naturally this ratio is much less in sweet cider or in distilled vinegar mixed with sweet cider than in cider vinegar. A certain amount of glycerol is also a characteristic of cider and wine vinegar as well as of the fermented liquors from which they are made. The analyses made by Ross, by Bender, and by Goodenow have established 0.24 per cent of glycerol as the minimum for generator vinegar.

The distinction of wine and malt vinegars from other kinds is not so important in the United States as in Europe. Wine vinegar is characterized by the presence of tartaric acid, free and combined as cream of tartar (potassium bitartrate) or in other combination, whereas the non-volatile acid of cider vinegar is largely malic. Malt vinegar usually contains more solids and phosphoric acid than cider or wine vinegar and is also characterized by the presence of dextrin and maltose. Glucose vinegar and molasses or sugar vinegar are relatively of small importance. The former is dextro-rotatory both before and after inversion, the latter is dextro-rotatory before but levorotatory after inversion. Cider vinegar is invariably levorotatory.

\*Determination of Solids and Acidity in Vinegar. See pp. 166 and 168. The determination of other constituents need not be taken up in this short course. They are of interest chiefly in food inspection.

#### VARIOUS FRUIT PRODUCTS

A great variety of food products formerly prepared only in the household are now made and put up in suitable containers in large establishments. Among the best known are canned fruits, dried fruits, preserves, jellies, catsup, and mince-meat.

Methods of Analysis. The products named may be analyzed by the methods described in Chapter IV and on the foregoing pages of this chapter, introducing slight modifications when needed.

Preservatives. Only two chemical preservatives are now used to any considerable extent in fruit products made in the

United States. These are, (1) Sulphur Dioxide, employed in bleaching as well as preserving dried fruits, and (2) Sodium Benzoate, added to preserves, jellies, catsup, and mince-meat.

Sulphur dioxide is determined by the method described for meat products containing sulphites (p. 38), sodium benzoate by Dunbar's modification of the La Wall and Bradshaw method. The latter method depends on the extraction of the benzoic acid by chloroform after adding common salt to hold back certain interfering substances. The benzoic acid is weighed and in addition may be titrated in an alcoholic solution.

### CHAPTER IX

#### FLAVORING EXTRACTS

Foop, in the restricted sense, includes only such products as furnish the body with materials for the production of muscular energy, heat, or the repair of tissues; in the broader sense it includes products used solely for their flavor, such as spices and flavoring extracts, or for their flavor and stimulating properties, such as tea and coffee. Proteins and fat in a state of purity have little or no flavor and the same is true of starch and dextrins of the carbohydrate group. Sugars are the exception among the nutritive substances in that they have pronounced flavors. The flavor of most natural or manufactured foods is due to minor constituents produced in the animal or vegetable organism or else developed by roasting or other method of preparation. When the flavor is lacking or needs modifying, spices, extracts, or similar materials are used.

Distinction of Spices from Extracts. Spices are natural products used solely for their flavoring constituents. Although they consist chiefly of the substances belonging to the six groups considered in Chapter IV, their flavoring power, due to minor constituents, is so great that they are used in quantities too small to aid appreciably in nutrition. The valuable ingredients are essential oils and other pungent or aromatic bodies. Often the flavor is a blend resulting from the presence of two or more constituents. Most flavoring extracts are alcoholic solutions (tinctures) of essential oils such as oil of lemon, orange, almond, clove, cinnamon, nutmeg, peppermint, or wintergreen. Vanilla extract, however, contains vanillin, a crystalline substance, and various other aromatic substances derived by direct extraction of the dried fruit.

Nature of the Analytical Methods. In the determination of the essential oil in lemon and orange extract a centrifugal method, employing the Babcock apparatus, and a polariscopic method, involving the technique described in the chapter on sugars (p. 131), are used. Vanillin and coumarin are determined gravimetrically in vanilla extract and substitutes by extraction with immiscible solvents and vanillin is also estimated colorimetrically. Citral, one of the flavoring constituents of lemon and orange extract, is also determined by a colorimetric method. The analysis of extracts accordingly furnishes varied experience and the methods are typical of many others devised for the analysis of various materials including, not only foods but drugs and other technical products. The experience of carrying on two processes at the same time is also valuable.

In this connection it should be reiterated that the purpose of this book is not to describe a great number of tedious processes regardless of variety or importance, but rather a carefully selected number illustrative of types, striving at the same time to give a general idea of the subject of food analysis and the composition of foods. Too often the student is staggered by page after page of dry description and fails to grasp the subject as a whole or to appreciate its absorbing interest and practical importance.

### VANILLA EXTRACT AND SUBSTITUTES

Vanilla Beans. The term bean is a misnomer, as the product is not the seed of a legume, but the fruit of an orchid (Vanilla planifolia). The narrow pods when taken from the plant are green and about the size and length of a lead pencil, but on drying become black and much shriveled (Fig. 102). They contain great numbers of black seeds so minute that they form a powder. The world' supply comes chiefly from Mexico, the insular possessions of France off the coast of Africa (Bourbon or Réunion, Madagascar, etc.), South America, and Tahiti, the market value diminishing in the order named. The better sorts sell for several dollars a pound. They contain, according to analyses by Winton and Berry, from 1.50 to 3.50 per cent of

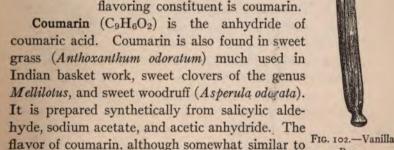
vanillin, also other aromatic constituents not yet isolated which,

although present in small amount, contribute materially to the delicate flavor.

Vanillin (C8H8O3) is the methyl ether of protocatechuic aldehyde. It may be obtained as white crystalline needles either by extraction of vanilla beans with ether or other solvents, or synthetically by the oxidation of the eugenol of oil of cloves with alkaline potassium permanganate. The synthetic product has sold as low as 35 cents per ounce, whereas if made from vanilla beans it would cost ten to twenty times that amount. In other words only from onetenth to one-twentieth of the cost of vanilla beans can properly be attributed to their vanillin

> content, the remainder being paid for the other flavoring constituents.

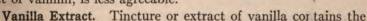
> Tonka Beans (Fig. 103) are the seeds of a tree (Dipterix odorata) native to Guiana. As the tree belongs to the Leguminosæ the seeds are appropriately termed " beans." They resemble almonds in size and shape. The chief



that of vanillin, is less agreeable.

Fig. 103.-Tonka

Bean.





ingredients of the vanilla bean soluble in 60 per cent alcohol and added cane sugar. It is, therefore, quite complicated in its composition and belongs in a different class from most flavoring extracts such as almond, peppermint, wintergreen, cinnamon, cassia, cloves, and nutmeg, which are merely alcoholic solutions of essential oils. In addition to vanillin, vanilla extract contains brown coloring matter and other substances forming a flocculent precipitate with normal lead acetate solution, resin, organic acid, and certain ash constituents.

The formula of a former edition of the United States Pharmacopæia for the preparation of vanilla extract is as follows:

"Vanilla, cut into small pieces and bruised, 100 grams.

"Sugar, in coarse powder, 200 grams.

"Alcohol and water, each, a sufficient quantity to make 1000 CC.

"Mix alcohol in the proportion of 650 cc. of alcohol to 350 cc. of water. Macerate the vanilla in 500 cc. of this mixture for twelve hours, then drain off the liquid and set it aside. Transfer the vanilla to a mortar, beat it with the sugar into a uniform powder, then pack it in a percolator, and pour upon it the reserved liquid. When this has disappeared from the surface. gradually pour on the menstruum, and continue the percolation, until 1000 cc. of tincture are obtained."

Vanilla extract prepared according to this formula varies according to Winton and Berry between the following limits:

Vanillin, 0.10 to 0.35 gram per 100 cc.

Normal lead number, 0.40 to 0.80.

Per cent of color in lead filtrate, not more than 10 per cent red or 12 per cent yellow.

Ratio of red to yellow in the extract, not less than I: 2.2.

Color insoluble in amyl alcohol, not more than 40 per cent.

The range in acidity and ash was found by Winton, Albright, and Berry to be as follows:

Total acidity, 30 to 52 cc. N/10 alkali per 100 cc.

Acidity, other than vanillin, 14 to 42 cc. N/10 alkali per 100 cc.

Total ash, 0.220 to 0.432 gram per 100 cc.

Substitutes for Vanilla Extract. Synthetic vanillin, tonka beans, and synthetic coumarin are much used in the preparation of flavoring solutions designed as substitutes for or imitations of vanilla extract. As both vanillin and coumarin are colorless, caramel is commonly added to these solutions in sufficient amount to impart a deep coffee color to the liquid.

Such preparations, although often containing percentages of vanillin within the limits for vanilla extract, are characterized by their low normal lead number, low acidity other than vanillin, low ash, low ratio of red to yellow color in the extract, high percentages of color, both in the lead filtrate and insoluble in amyl alcohol. Coumarin, which is absent in vanilla extract, is often present.

\*Materials for Laboratory Practice. The analysis of a genuine vanilla extract and a substitute, consisting of a solution of vanillin and coumarin colored with caramel, will give the student sufficient experience for an understanding of the most important methods and the interpretation of results. The vanilla extract can either be prepared in the laboratory according to the U. S. P. formula or may be obtained of a reputable manufacturer. The substitute may be prepared by dissolving 2 to 4 grams of vanillin, 0.4 to 1.0 gram of coumarin, and 200 grams of sugar in a mixture of equal parts of 95 per cent alcohol and water, adding sufficient caramel to impart a deep coffee color, and making up to 1 liter with the same menstruum. Care should be taken that the amount of caramel added is not sufficient to impart a color too deep to be conveniently measured by the Lovibond tintometer.

The composition of the vanilla extract can be learned only by analysis, whereas the precentages of vanillin and coumarin in the substitute will be known, at least to the instructor, from the quantities used. At least 57 cc. of each preparation should be available for each student, 50 cc. for the gravimetric analysis, 5 cc. for the volumetric determination of vanillin, and 2 cc. for determining the color value.

Only single determinations need be made on each material by the methods described. The results for vanillin by the two methods should check each other, thus serving as duplicates, and the single gravimetric analysis in the case of the vanilla extract will demonstrate the absence of coumarin quite as well as duplicates. The estimation of the color value involves such simple manipulation as to preclude the probability of error. The single result for coumarin in the vanilla substitute and the single results for normal lead number in both materials ordinarily would require checking, if only a single analyst were involved, but for our purpose a comparison of the results of the different students will suffice.

It may here be reiterated that agreeing results by the same analyst are often not conclusive, as he is liable to make the same error in both determinations. In important work it is desirable that the duplicates be made by different analysts and if possible with different reagents and apparatus, thus eliminating the personal equation.

\*Determination of Vanillin and Coumarin by the Modified Hess and Prescott Method. This process, in its original form devised by Hess and Prescott, has been modified by the author, collaborating with Silverman, Bailey, Lott, and Berry, in order to prevent loss of coumarin, detect the presence of acetanilide (formerly much used as an adulterant of vanillin), and permit the determination of normal lead number in the same weighed portion.¹ It depends on the principle that ammonia water, acting on the ether solution of vanillin and coumarin, forms with the aldehyde vanillin a compound soluble in water, but does not affect the coumarin, which remains in solution in the ether.

Extraction with Immiscible Solvents. This method is particularly instructive, as it is a type of numerous methods involving the extraction of one or more constituents from an aqueous

<sup>&</sup>lt;sup>1</sup> Jour. Amer. Chem. Soc., 1899, 21, p. 256; U. S. Dept. Agr., Bur. Chem., Bul. 152, p. 147

liquid with an immiscible solvent such as ether, chloroform, or carbon bisulphide. Various forms of apparatus for the continuous extraction of one liquid with another have been devised, but for ordinary purposes shaking in a separatory funnel, as here described, is preferable. Care must be taken to avoid too violent shaking with ether, as otherwise an emulsion will be formed which is not easily broken up. The ether should always be poured out of the neck of the funnel after drawing off the

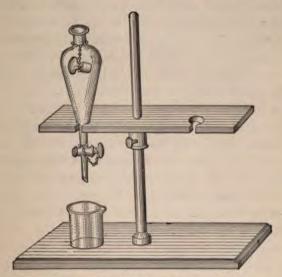


Fig. 104.—Squibb Separatory Funnel.

aqueous liquid through the stopcock, thus obviating contamination with the water-soluble constituents such as the sugar of the extract or the ammonium chloride formed by the neutralization of ammonium hydroxide.

Fig. 104 shows the pear-shaped or Squibb form of separatory funnel, which is well adapted for the analysis of vanilla extract. The support has holes with slots for inserting the separatory funnels, but may be used also for ordinary funnels.

Process. Pipette 50 cc. of the extract directly into a tared 250-cc. beaker with marks made with diamond ink showing vol-

umes of 80 and 50 cc.; dilute to 80 cc. with water boiled until free from carbon dioxide, and evaporate to 50 cc. in a pan of water kept at 70° C. by a Bunsen burner. Dilute again to 80 cc. and evaporate to 50 cc. as before. Transfer to a 100-cc. graduated flask, rinsing the beaker with hot carbon dioxide-free water, taking care not to use more than 25 cc.; add 25 cc. of standard lead acetate solution (80 grams of chemically pure crystallized lead acetate dissolved in water and made up to 1 liter), make up to the mark, shake, and place in a bacteriological incubator, in a water bath provided with a thermostat, or in other suitable apparatus, kept at a temperature of from 37° to 40° C.

Two laboratory periods of four hours each will be required for the work up to this point. During the first of these periods, while the dealcoholizing is proceeding, there will be time to determine vanillin by the Folin and Denis method (p. 192). The color value of the vanilla and vanilla substitute (p. 189) can be determined during the second period; the glass dishes for the vanillin and the coumarin can also be weighed.

On the third day, after the flask has been kept at 37° to 40° for eighteen to twenty hours, filter through a small dry filter and pipette off 50 cc. of the filtrate into a Squibb separatory funnel of 125 cc. capacity.

For the determination of normal lead number, pipette off 10 cc. of the filtrate into a beaker and precipitate as described on p. 191. Use the remainder of the lead filtrate for the determination of color value (see p. 190). This can be carried out while the vanillin and coumarin are being shaken out with ether. If kept until the next day a cloudiness, due to absorption of carbon dioxide and precipitation of lead carbonate, is liable to appear.

To the 50 cc. of the filtrate in the separatory funnel add 20 cc. of ether and shake cautiously several times. Draw off carefully through the stopcock the aqueous liquid, together with any ether emulsion, and then pour the clear ether solution from the mouth into a beaker. Return the aqueous solution to the separatory funnel and shake out as before using, however,

15 cc. of ether. Repeat this treatment twice. Reject the extracted aqueous liquid and rinse the separatory funnel.

Pour the combined ether solutions into the rinsed separatory funnel, add 10 cc. of 2 per cent ammonium hydroxide solution, and shake several times. Draw off the ammoniacal solution into a beaker, taking care not to allow any of the ether solution to pass through with it. Shake out with three more portions of the ammonium hydroxide solution exactly as before, except that 5 cc. are used.

Transfer the ether solution, containing the coumarin if present and from which the vanillin has been removed in the

ammoniacal solution, to a weighed lowform, glass crystallizing dish, 60 mm. in diameter, with an etched circle on which is placed an identification mark with a lead pencil (Fig. 105).

Add to the combined ammoniacal solutions with stirring 10 per cent hydrochloric acid until it is slightly acid to test paper. This should be done without

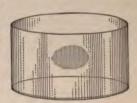


Fig. 105.—Crystallizing Dish.

delay, as the ammoniacal solution on standing grows slowly darker with a loss of vanillin. Cool, transfer to the separatory funnel and shake out the vanillin with four portions of ether, as described for the first ether extraction, removing the ether solution each time to a weighed crystallizing dish. Allow the ether in this crystallizing dish, as well as that in the crystallizing dish containing the ethereal solution of the coumarin, to evaporate at room temperature until the next day. Do not attempt to hasten the evaporation of the ether by heating or by an air current, as this will cause condensation of moisture in the dishes, owing to the lowering of the temperature.

On the following day (the fourth of the work on extracts), place the crystallizing dishes in a sulphuric acid desiccator, then finish the determination of normal lead number as described on p. 191.

On the fifth day weigh the dishes containing the vanillin

and coumarin and calculate the weight per 100 cc. of each. The genuine vanilla extract will, of course, contain no coumarin, although a very small amount of resinous material may be obtained which, were it crystalline and present in considerable amount, would be considered coumarin.

In the vanilla substitute the crystals of coumarin are recognized by their needle shape and characteristic odor. Determine the melting-point as described below and subject the remainder to the Leach test (p. 189). The identification of the coumarin is essential, as its presence constitutes an adulteration in a preparation purporting to be pure vanilla extract. At one time synthetic vanillin was often adulterated with acetanilide, which, by the process above described, would be largely weighed with the coumarin but could be subsequently separated.

\*Determination of the Melting-point of Coumarin. The melting-point, so often determined in the organic laboratory, serves for the identification of many crystalline substances.

The required apparatus is the same as that used for determining the melting-point of fats (Fig. 96), except that the flask and tube are smaller and the capillary tubes are closed at the lower end. Instead of water, concentrated sulphuric acid is used.

Introduce a crystal or two of the substance into a capillary tube closed at one end and place this against the bulb of the thermometer where it adheres owing to the viscosity of the acid. Slowly heat the acid in the flask over a Bunsen burner. The heat is communicated to the acid in the inner tube and finally to the substance in the capillary tube. Note the temperature at which the crystal melts.

To make capillary tubes suitable for melting-point determinations, heat the middle part of a test-tube and draw down to the size of the lead in a pencil, cut into lengths of about  $1\frac{1}{2}$  in., and close one end of each, by fusing.

The melting-point of pure coumarin is 67° C. That obtained in the analysis may melt slightly below that temperature, as any impurity depresses the melting-point. The variation from 67° should, however, not be more than a degree or two. Leach Test for Coumarin. To the portion of the crystals remaining in the crystallizing dish (p. 188), add a few drops of water, warm gently, and add a few drops of a solution of iodine in potassium iodide. In the presence of coumarin a brown precipitate will form which, on stirring with the rod, will soon gather in dark-green flocks.

\*Determination of Color Value of Vanilla Extract and Sub-

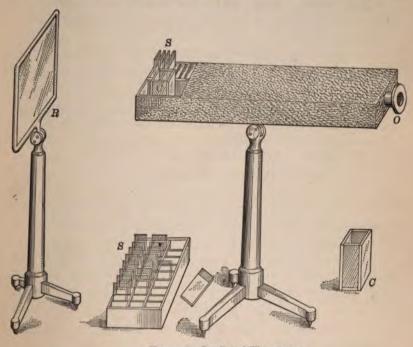


Fig. 106.—Lovibond Tintometer.

stitutes. The Lovibond Tintometer (shown in Fig. 106), is a simple instrument so arranged that light is reflected from a square of opal glass R, through a cell with glass sides C, containing the liquid under examination, and at the same time through standard colored glass slides S, added, one by one, to a carrier until the colors, as seen through an eyepiece O, match.

The standard slides used in general work are red, yellow, and

blue in even graduation from .006 to 20 tint units, which can be combined so as to produce any desired tint or shade of any color. The results are expressed in terms of standard dominant colors (red, yellow, and blue), subordinate colors (orange, green, and violet), obtained by combining equal values of two dominant colors, and neutral tint (black), obtained by combining equal values of the three dominant colors.

Thus 
$$0.6R + 5.6Y = 0.6O + 5.0Y$$
,  
 $0.08R + 1.5Y + 0.2B = 0.08N + 0.12G + 1.3Y$ ,  
 $1.2R + 1.0B = 1.0V + 0.2R$ 

in which R = red, Y = yellow, B = blue, O = orange, G = green, V = violet, and N = neutral tint or black.

For vanilla extract work only the following 17 slides are needed:

It is recommended, however, that a set of blue slides of the denominations given for the red be provided for the highly instructive study of colors and color combinations.

Process. Pipette 2 cc. of the extract into a 50-cc. graduated flask and make up to the mark with a mixture of equal parts of 95 per cent alcohol and water. Determine the color value of this diluted extract in terms of red and yellow by means of a Lovibond tintometer, using the 1-in. cell. To obtain the color value of the original extract multiply the figures for each color by 25.

For example, a reading of 0.6 red and 2.1 yellow obtained on the diluted extract corresponds to a color value of 15 red and 52 yellow calculated to the original extract.

\*Determination of Residual Color after Precipitation with Lead Acetate. As soon as possible after filtration determine the color value, in terms of red and yellow, of the filtrate from the lead acetate precipitate, obtained in the determination of vanillin and coumarin (p. 186), using the 1-in. Lovibond cell. Multiply the reading by 2, thus reducing the result to the basis of the original extract.

In case the actual reading of the solution is greater than 5 red and 15 yellow, as may happen if the extract is highly colored with caramel, the  $\frac{1}{2}$ - or  $\frac{1}{4}$ -in. cell should be employed and the readings multiplied respectively by 4 or by 8; or else 10 cc. of the solution should be diluted to 50 cc. in a graduated flask, mixed, examined in the 1-in. cell, and the reading multiplied by 10.

Divide the figures for red and yellow respectively, by the corresponding figures of the original extract and multiply the quotients by 100, thus obtaining the percentages of the two colors remaining in the lead acetate filtrate.

For example, if the color value of the original extract is 15 red and 52 yellow and the color value of the lead acetate filtrate, also measured in the 1-in. cell, is 0.6 red and 2.4 yellow, then the residual color, after precipitation with lead acetate, calculated to the basis of the original extract, is 1.2 red and 4.8 yellow or 8 per cent of the red and 9.2 per cent of the yellow.

\*Determination of Normal Lead Number by the Winton and Lott Method. Mix the 10 cc. aliquot of the filtrate from the lead acetate precipitate, obtained in the determination of vanillin and coumarin (p. 186), with 25 cc. of water, boiled until free from carbon dioxide, and a moderate excess of sulphuric acid. Add 100 cc. of 95 per cent alcohol and mix again.

Let stand overnight, collect the lead sulphate on a weighed Gooch crucible, wash with six portions of 95 per cent alcohol, filling the crucible each time and allowing it to empty before adding the next portion, dry at a moderate heat on a piece of asbestos paper, ignite at low redness for three minutes, taking care to avoid the reducing flame, cool, and weigh. The normal lead number is calculated by the following formula:

$$P = \frac{100 \times 0.6831(S - W)}{5} = 13.662(S - W),$$

in which P=normal lead number, S=grams of lead sulphate corresponding to 2.5 cc. of the standard lead acetate solution as determined in blank analyses, and W=grams of lead sulphate obtained in 10 cc. of the filtrate from the lead acetate precipitate as above described.

The standard of the lead acetate solution is determined by blank analyses and does not change appreciably on standing in a well-stoppered bottle. The beginner probably will not find time to determine the standard and can accept the figures obtained by the instructor or the more advanced student.

The normal lead number of the genuine extract should vary between the limits given (p. 182), while that of the substitute will be practically zero.

\*Determination of Vanillin by the Folin and Denis Method. This method<sup>1</sup> is based on the fact that vanillin (as well as other mono-, di-, and tri-hydric phenol compounds), when treated in an acid solution with phosphotungstic-phosphomolybdic acid, gives on addition of an excess of sodium carbonate, a beautiful deep blue color. It yields accurate results, requires but 5 cc. of the material, and is exceedingly rapid. An analyst familiar with the process can make ten or twelve determinations in an hour, whereas, working under favorable conditions, he would not be able to make the same number of determinations by the Hess and Prescott method in less than three days. For inspection purposes the latter method has the advantage that the vanillin and coumarin are obtained in crystalline form for subsequent tests; furthermore coumarin, normal lead number, and color value of the lead filtrate are determined in one weighed portion.

Given the reagents, the student will have no difficulty in making determinations of vanillin in the practice samples (p. 183), by the Folin and Denis method, while waiting for the dealcoholizing required in the Hess and Prescott method. It may here be mentioned that it is often necessary for the analyst, in order to use his time to the best advantage, not only to carry

<sup>1</sup> Jour. Ind. Eng. Chem., 1912, 4, p. 670.

along together determinations by the same method on different samples, but also in the intervals to have in progress analyses by entirely different methods.

Nature of Colorimetric Methods. The Folin and Denis method is typical of numerous colorimetric methods in that it depends on the formation of a colored compound with the substance to be determined, the amount present being estimated from the intensity of the coloration of the solution as compared with that of a solution containing a known amount of that

substance treated in the same manner. The solution of the unknown may either be compared with several solutions, prepared with different amounts of a standard solution, selecting for the calculation the one that matches in shade, or else it may be compared with a single solution, varying the height of the column of one or the other until the colors reflected through the two columns match and calculating the result by the rule of three. The former procedure is used in determining the free and albuminoid ammonia in potable water by a process known as "Nesslerizing," while the latter is more commonly employed in food analysis. The comparison of the solution of the unknown with the standard may be made in two tubes, each provided with a stopcock at the bottom whereby a portion of the darker solution may be drawn off until

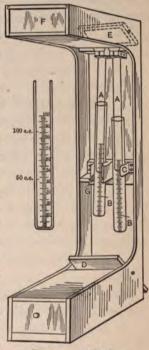


Fig. 107.—Schreiner Colorimeter.

the two columns match in tint or else a colorimeter may be used.

The Schreiner Colorimeter is well adapted for our purpose, being inexpensive, of simple construction, and accurate. This apparatus, shown in Fig. 107, consists of two graduated tubes

B, containing the standard and unknown colorimetric solutions, the height of the column of liquid in both tubes being changed by two immersion tubes A, which remain stationary while the graduated tubes are raised or lowered in the clamps C. The mirror D reflects the light through the tubes, and the mirror E reflects it again to the eye of the operator at F.

In making the comparisons the tube containing the solution of either known or unknown strength is set at a definite point, and the other tube is raised or lowered until the colors match. If R is the reading of the standard solution of the strength S, and r the reading of the colorimetric solution of unknown strength s, then

$$s = \frac{R}{r} S$$
.

If desired, standard slides of colored glass, such as accompany the Lovibond tintometer, may be used at G for matching the solution of unknown strength, the value of these slides being determined by comparison with a standard solution.

Suggestions. The student should not be discouraged if at first he has difficulty in securing concordant readings in the comparison of the two solutions in the colorimeter. Some experience is required before the eye can detect slight differences in shade and arrive at the exact point where the two solutions match in color intensity. The following hints may prove helpful:

Choose a soft but sufficient light, best at a north window or reflected from the north sky; never use direct sunlight. Practice at first with the same solution in both tubes. Do not use too high columns, as it is difficult to match deep colors. Avoid straining the eyes; adjust the tubes rapidly until the colors match approximately, then look away and when the eyes have rested a moment make the final adjustment in about five seconds. Do not attempt colorimetric work when the light is poor, when the eyes are tired, or when you are hurried or otherwise mentally disturbed.

Reagents. (1) Standard Vanillin Solution. Dissolve 0.1 gram of pure vanillin in water and make up to 1 liter.

- (2) Phosphotungstic-phosphomolybdic Acid Reagent. To 100 grams of pure sodium tungstate and 20 grams of phosphomolydic acid (free from nitrates and ammonium salts) add 100 grams of syrupy phosphoric acid (containing 85 per cent H<sub>3</sub>PO<sub>4</sub>) and 700 cc. of water. Boil over a free flame for one and one-half to two hours, cool, filter, if necessary, and make up with water to 1 liter. An equivalent amount of pure molybdic acid may be substituted for the phosphomolybdic acid.
- (3) Sodium Carbonate Solution. Prepare a solution of the c.p. salt, saturated at room temperature.
- (4) Lead Solution. Dissolve 50 grams each of basic and neutral lead acetate in water and make up to 1 liter.

Process. Pipette 5 cc. of the extract or substitute into a graduated 100-cc. flask, add about 75 cc. of cold tap water and 4 cc. of lead-solution, make up to the mark with water and shake. Filter rapidly through a folded filter paper and pipette 5 cc. of the filtrate, corresponding to 0.25 cc. of the extract, into a 50-cc. graduated flask. Into another 50-cc. graduated flask pipette 5 cc. of the standard vanillin solution, which volume contains 0.0005 gram of vanillin. To each flask add from a pipette 5 cc. of the phosphotungstic-phosphomolybdic reagent, directing the stream against the neck in such a manner as to wash down any adhering vanillin. Shake the flasks by a rotary motion, allow to stand for five minutes, then fill to the mark with saturated sodium carbonate solution. Thoroughly mix the contents of the flasks by inverting several times and allow to stand for ten minutes in order that the precipitation of sodium phosphate may be complete. Filter rapidly through folded filters and compare the color of the deep-blue solutions, which must be clear, in the colorimeter.

In this, as in all colorimetric methods, a slight cloudiness of the solution of the unknown, by cutting off more light than the standard, gives a low reading and correspondingly high result. Calculate the grams of vanillin per 100 cc. as follows:

$$P = \frac{0.0005R \times 100}{0.25r} = \frac{R}{5r},$$

in which P is the grams of vanillin per 100 cc., R is the reading of the standard solution and r is the reading of the unknown solution in the colorimeter.

Determination of Other Constituents. Sucrose is calculated from the polariscopic readings (p. 133), and Alcohol from the specific gravity of the distillate obtained by direct distillation as in the case of a liquor (p. 172). The amount of neither of these constituents throws any light on the genuineness of an extract; on the other hand, the percentage of Ash and the Acidity other than vanillin, as shown by Winton, Albright, and Berry, bear a striking relation to the normal lead number and are valuable in distinguishing genuine vanilla extract from solutions of vanillin and coumarin. The solubility and alkalinity of the ash serve to detect the presence of added alkali in vanilla extract.

The total acidity is determined by titration, using phenolphthalein as an indicator, the acidity due to vanillin by calculation from the percentage of that constituent. Total ash is determined by evaporation and incineration at a dull red heat.

#### LEMON EXTRACT

Lemon Oil is the essential oil obtained from lemon peel. The chief regions of production are Sicily and adjoining parts of the Italian mainland, where the manufacture of oil from the peel and citrate of lime from the pulp are carried on in the same factories. Commercial citric acid is obtained by heating citrate of lime with sulphuric acid.

Limonene ( $C_{10}H_{16}$ ), a dextro-rotatory terpene with a strong flavor, makes up about 90 per cent of lemon oil; Citral ( $C_{10}H_{16}O$ ), an aldehyde with a delicate flavor and the characteristic odor of lemon peel is present to the extent of 4 to 5 per cent. Both

of these substances occur in the peel of other citrus fruits, and citral is also present in lemon grass. Commercial citral is obtained from lemon grass or artificially by the oxidation of geraniol. Limonene is soluble in strong alcohol, but insoluble in dilute alcohol, while citral is soluble in both.

Lemon Extract as recognized by Federal and State standards, as well as by the trade, is a solution in strong alcohol of at least 5 per cent by volume of lemon oil, with or without the coloring matter and other extractive substances of lemon peel. When diluted with water, it becomes cloudy, due to the precipitation of the limonene. The flavor, aside from that of the alcohol, which evaporates in cooking or is lost by dilution, is a combination of the strong taste of limonene and the delicate aroma of citral.

Terpeneless Lemon Extract is a solution prepared by shaking lemon oil with dilute alcohol or dissolving so-called terpeneless lemon oil in that solvent and should contain at least 0.2 per cent, by weight, of citral. It is used for flavoring soda water and other liquids to which lemon extract would impart a turbidity. As under ordinary market conditions the cost of a lemon extract is due more to the alcohol than to the lemon oil, the cheaper terpeneless extract is often sold for family use and has not always been labeled so as to show its true character.

\*Material for Laboratory Practice. A lemon extract containing from 5 to 8 per cent of lemon oil dissolved in 95 per cent alcohol and a terpeneless extract containing 0.20 to 0.30 per cent of citral, prepared by dissolving 1 gram of terpeneless lemon oil in 300 cc. of 50 per cent alcohol, are suited for analytical practice. These may either be prepared in the laboratory or obtained from the manufacturer or grocer.

Dilute a portion of the terpeneless extract with an equal volume of water. No cloudiness should appear, showing that lemon oil is not present and that a quantitative determination of this substance is unnecessary.

Arrangement of Time. On the day when the vanillin and coumarin, obtained in the analysis of vanilla extract and sub-

stitute, are weighed (p. 187), time will be found to determine lemon oil by Mitchell's two methods <sup>1</sup> in the lemon extract. As the polarization method involves no manipulation other than direct polarization and the centrifugal method serves as a check, a single determination by each method will suffice.

Citral can be determined in both samples on the following day, after the extraction of caffeine from coffee has been started (p. 205).

\*Determination of Lemon Oil by the Mitchell Polariscopic Method. Polarize the extract, without dilution, in a 200-mm. tube in the same manner as is described in Chapter VI. Divide the reading obtained in degrees Ventzke on the sugar scale by the factor 3.2. If sugar or other optically active substances are not present, as is almost always the case, the quotient will be the per cent of lemon oil by volume.

\*Determination of Lemon Oil by the Mitchell Centrifugal Method. Pipette 20 cc. of the extract into a Babcock milktest bottle (p. 19), add 1 cc. of dilute hydrochloric acid (1:1) and 25 to 28 cc. of water previously warmed to 60° C., mix, and let stand in water at 60° C. for five minutes, whirl in a centrifugal machine five minutes, as in milk analysis, fill with water at 60° nearly to the 10 per cent graduation, and whirl again for two minutes. Immerse in water at 60° nearly to the top of the neck for a few minutes and finally read the length of the column exactly as in the Babcock test. When the result is over 2 per cent add 0.4 per cent to correct for lemon oil retained in the solution, when less than 2 per cent but more than 1 per cent, add 0.3 per cent. The result thus corrected should agree with that by the polarization method within 0.2 per cent.

A marked disagreement by the two methods would indicate the presence of a foreign essential oil, such as oil of citronella, in which case the oil layer obtained in the test bottle should be examined as to its refractive index and other properties. Fortunately such addition is rarely, if ever, practiced.

\*Determination of Citral by the Hiltner Method. This

1 Jour. Amer. Chem. Soc., 1899, 21, p. 1132.

colorimetric method <sup>1</sup> measures the strength of terpeneless lemon extracts and also detects the substitution in lemon extract of "washed lemon oil," the residual oil after shaking with dilute alcohol in the manufacture of terpeneless extracts, for natural lemon oil. A lemon extract made from washed lemon oil will naturally be deficient in citral.

Reagents: (1) Metaphenylene Diamine Hydrochloride Solution. Prepare a 1 per cent solution in 50 per cent ethyl alcohol. Decolorize by shaking with fuller's earth or animal charcoal, and filter through a double filter. The solution should be bright and clear, free from suspended matter, and practically colorless. It is well to prepare only enough for the day's work, as it darkens on standing. The color may be removed from old solutions by shaking again with fuller's earth. This reagent gives a yellow color with citral but no appreciable color with the other aldehydes present in lemon extract or lemon oils.

- (2) Standard Citral Solution. Dissolve 0.25 gram of c.p. citral in 50 per cent ethyl alcohol and make up the solution to 250 cc.
- (3) Alcohol. For the analysis of lemon extracts 90 to 95 per cent alcohol should be used, but for terpeneless extracts 40 to 50 per cent strength is sufficient. Filter to remove any suspended matter. If not practically colorless, render slightly alkaline with sodium hydroxide and distill. Purification from aldehyde is unnecessary.

Process. All the operations are carried out at room temperature. Weigh into a 50-cc. graduated flask 25 grams of the extract, make up to the mark with alcohol, and mix thoroughly. This diluted extract can be used by the whole class. Pipette into a 50-cc. graduated flask 2 cc. of the diluted extract (equivalent to 1 gram of the original extact), add 10 cc. of metaphenylene diamine hydrochloride solution, make up to the mark with alcohol, and shake. Into another 50-cc. flask pipette 2 cc. of the standard citral solution (containing 0.002 gram of citral), add 10 cc. of metaphenylene diamine hydrochloride solution, make

<sup>1</sup> Jour. Ind. Eng. Chem., 1909, 1, p. 798.

up to the mark with alcohol, and shake. Compare at once the color of the two solutions in the Schreiner colorimeter (p. 193). Calculate the result by the following formula:

$$P = \frac{0.002R \times 100}{r} = \frac{0.2R}{r},$$

in which P is the per cent by weight of citral, R is the reading of the standard solution, and r is the reading of the unknown solution in the colorimeter.

Determination of Other Constituents. Alcohol is determined in a portion of the extract from which lemon oil has been removed by dilution, shaking with magnesium carbonate, and filtration. The oil which is precipitated by dilution is mechanically held by the magnesium carbonate, thus affording a clear filtrate. The alcohol is obtained from an aliquot of the filtrate by distillation and its amount calculated from the specific gravity.

Total Aldehydes are estimated by a method devised by Chace, depending on the amount of color developed in a solution of the dye fuchsin, which has been decolorized by sulphur dioxide. Practically all the aldehyde content of lemon extract or terpeneless lemon extract is citral. Other aldehydes are present in orange extract. Neither the alcohol nor the total aldehydes need be determined by the student.

Coloring Matter if of coal-tar origin is detected by the usual methods, if from lemon peel by Albrech's method.

Analysis of Other Extracts. Orange Extract is analyzed by practically the same methods as lemon extract.

Denis and Dunbar <sup>1</sup> have devised a method for the determination of benzaldehyde, the chief constituent of *Almond Extract*, based on its precipitation as hydrozone with phenyl hydrazine. Hortvet and West <sup>2</sup> oxidize it to benzoic acid.

Wintergreen oil is determined in Wintergreen Extract by the Hortvet and West method <sup>2</sup> depending on its conversion first

<sup>1</sup> Jour. Ind. Eng. Chem., 1909, 1, p. 256.

<sup>2</sup> Ibid., p. 84.

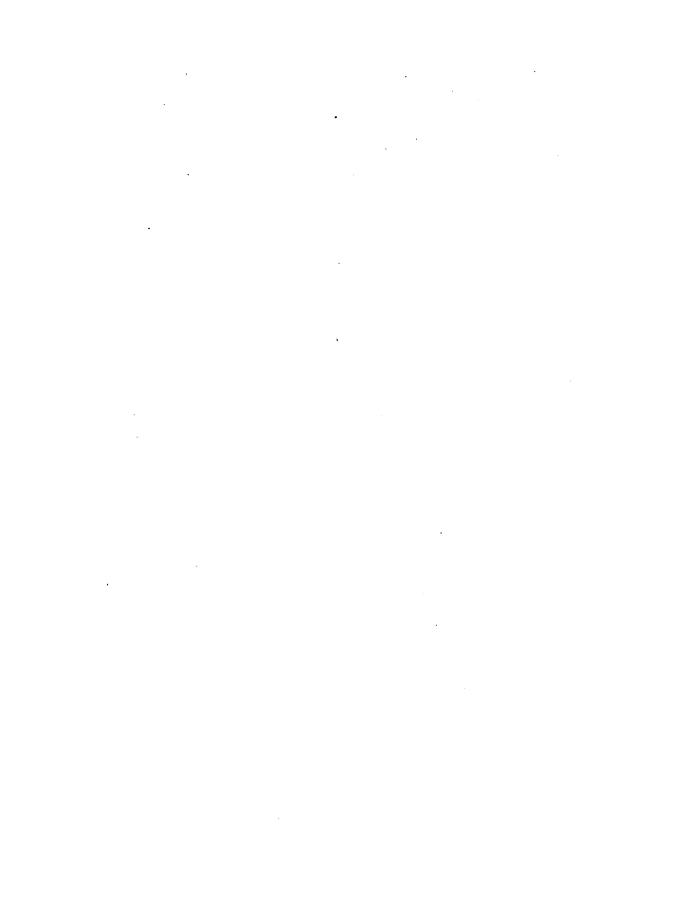
into potassium salicylate by boiling with potassium hydroxide and hydrogen peroxide solution and finally into salicylic acid by treatment with hydrochloric acid.

Peppermint oil is determined in *Peppermint Extract* by C. D. Howard's modification <sup>1</sup> of Mitchell's centrifugal method.

The essential oils in various *Spice Extracts* are estimated by methods devised by Hortvet and West <sup>2</sup> and C. D. Howard.<sup>1</sup>

<sup>&</sup>lt;sup>1</sup> Jour. Amer. Chem. Soc., 1908, 30, p. 608.

<sup>&</sup>lt;sup>2</sup> Jour. Ind. Eng. Chem., 1909, 1, p. 84.



### CHAPTER X

### COFFEE, TEA, AND COCOA

Food Value of Alkaloidal Beverages. Coffee and tea are valuable solely because of their flavoring and stimulating properties. A cup of either beverage has practically no food value except what is due to added milk, cream, and sugar. The quantity of the material used per cup is itself small and of this only a portion goes into solution in the water with which it is boiled or steeped; the remainder contained in the coffee grounds or spent tea leaves is rejected.

Chocolate, as such, and after removal of a portion of the fat, in which form it is known as cocoa, on the other hand, is not merely a flavor and stimulant, but a concentrated food, rich in fat and protein and valuable also for its starch.

The Stimulating Principles of alkaloidal beverages, Caffeine and Theobromine, the former being present in all these, the latter only in cocoa and chocolate, have been shown by Emil Fischer to be purin derivatives, closely related to xanthine. Their structural formulæ, which follow, show them to be respectively tri- and di-methyl xanthine.

These bases are also grouped with the alkaloids. Formerly the principle of tea was known as theine, but more recently it has been shown to be identical with the caffeine of coffee. Caffeine is present in chocolate and cocoa in smaller amounts than theobromine.

It is a remarkable fact that these stimulants are associated with flavors which are particularly acceptable to the human race and that of the tens of thousands of other plants not containing a stimulant none yields an infusion that attracts the appetite like these three "cups that cheer."

Flavors by a strange psychological association lead man not only to the true elements of nutrition he needs, but also to the stimulants he craves.

The Microscopic Structure of the three products (pp. 118 to 122) should be kept constantly in mind in considering the analyses given in this chapter. The study of structure and chemical composition of natural vegetable products should always go hand in hand, one throwing light on the other.

#### COFFEE

Composition of Coffee. The following table is based on analyses by Lythgoe <sup>1</sup> obtained on roasted samples of Santos, Porto Rico, Rio, Mocha, and Java coffees:

	Average.	Maximum.	Minimum.
Moisture	2.16	3.44	1.26
Fat (petroleum ether extract	13.75	15.18	12.28
Crude fiber	13.03	14.75	11.02
Protein	12.00	13.75	10.50
Caffeine	1.20	1.34	1.10
Ash	4.03	4.38	3.74
Nitrogen-free extract	53.83	55 - 72	49.29
	100.00	******	********
Hot water extract 2	25.80	27.70	24.60

<sup>&</sup>lt;sup>1</sup> Technology Quarterly, 1905, 18, p. 236. Other constituents were also determined.

<sup>&</sup>lt;sup>2</sup> Calculated from the 10 per cent extract obtained by boiling one hour according to McGill's method.

From the above figures it will be seen that only about onequarter of the coffee was extracted by boiling with water, the remainder being of no value to the consumer.

In addition to the constituents given in the table coffee contains a tannic acid known as caffetannic acid. C. D. Howard found 11.17 per cent of this acid in a sample of Java and Mocha coffee and Shanley 9.47 to 9.96 per cent in samples of Java, Mocha, and Rio coffee.

Coffee Substitutes. Chicory, the root of a plant related to the dandelion, is frequently mixed with coffee, imparting a sweetish taste and a deep-brown coloration. It yields more extractive matter when boiled with water than coffee.

Other Substitutes are made by roasting Barley, Malt, Wheat, Rye, Peas, Figs, Dried Bananas, Dried Beet Root, and various other products.

Most of the substitutes sink when stirred with cold water, whereas coffee floats. Microscopic examination will usually disclose the nature of the material provided it has not been roasted beyond recognition. Cereal products, peas, and bananas are rich in starch; chicory, figs, bananas, and beet root are rich in sugar. Neither starch nor an appreciable amount of sugar is present in coffee.

\*Determination of Caffeine in Coffee by the Görter Method. Material for Laboratory Practice. Powder a sample of coffee so it will pass a 25-mesh sieve. On this material determine caffeine in duplicate. The work requires three laboratory periods of four hours each, but during the first period, after the extraction has been started, there will be sufficient time to determine citral in the samples of lemon and terpeneless lemon extract (p. 198).

No other analytical work need be done on coffee, tea, or cocoa, as most of the methods are those already used in the analysis of other products or are such as can be understood from a brief description.

Process.<sup>1</sup> Mix 11 grams of the powdered coffee with 3 cc.

<sup>1</sup> Liebig's Annalen, 1908, 358, p. 327.

of water, allow to stand for thirty minutes, and place in the inner tube of a Johnson extractor. Should the tube be too small to hold the moistened coffee, use proportionately less of both the coffee and water. Connect an extraction flask (not weighed) and pour through the coffee sufficient chloroform to penetrate the mass and half fill the flask. Extract, as described on p. 56, for three hours. At the end of the extraction or on the next day, evaporate off the chloroform from the flask, taking care to avoid too violent ebullition with consequent mechanical loss.

Treat the residue in the flask with 5- to 10-cc. portions of boiling water, filtering each time through a plug of cotton contained in the stem of a funnel into a 55-cc. graduated flask. Cool to room temperature, make up to the mark, mix by inverting several times and pipette off 50 cc. (equivalent to 10 grams of the coffee) into a 125-cc. separatory funnel.

Shake with four portions of 15 cc. each of chloroform, as described for vanilla extract (p. 186). As the chloroform, unlike ether, forms a layer below the aqueous liquid, it may be drawn off each time through the stopcock. Use for collecting the four portions of chloroform a weighed tinned lead, aluminum, porcelain, or glass dish, which can be kept at a gentle heat so that while shaking with one portion, the preceding portion can be evaporating. Finish the evaporation and dry in a boiling water oven for one hour, cool in a desiccator, and weigh. Repeat the heating for one hour and weigh again. If the weight is constant, calculate the percentage of dry residue, which should be practically pure caffeine. In very exact work the nitrogen in the residue should be determined and the caffeine calculated. using the factor 3.464, but for our purpose the result obtained from the weight of the residue is sufficiently accurate, provided due care has been taken in the manipulation.

Other Methods for the Analysis of Coffee. The methods for the determination of Water, Fat, Crude Fiber, Total Nitrogen, and Ash are those described in Chapter IV. The per cent of Nitrogen as Caffeine is obtained by multiplying the per cent of caffeine by the factor 0.2886. To obtain the per cent of Pro-

TEA 207

tein subtract the caffeine nitrogen from the total nitrogen and multiply by 6.25.

Caffetannic Acid is extracted by 90 per cent alcohol, precipitated with lead acetate, and weighed, after drying at 100° C. as lead caffetannate, by Krug's method.<sup>1</sup>

TEA

Composition of Tea. Koenig <sup>2</sup> has compiled the results of 158 analyses of tea by different chemists with the following results:

	Average.	Maximum.	Minimum
Moisture	8.46	11.97	3.93
Nonvolatile ether extract	8.24	15.15	3.61
Essential oil	0.68		
Crude fiber	10.61	15.50	8.51
Protein	24.13	38.65	18.19
Theine (caffeine)	2.79	4.67	1.00
Ash	5.93	8.03	4.10
Tannin	12.35	25.20	4.48
Nitrogen-free extract other than			
tannin	<b>26</b> .81	]	
	100.00		

Kenrick <sup>3</sup> in the analysis of 53 samples of Chinese, Japanese, and Indian teas found that from 23.37 to 38.53 per cent of solids were extracted by a ten-minute infusion.

Coloring and Facing. Formerly green tea was colored by a blue pigment such as Prussian blue, ultramarine, or indigo, often with the addition of turmeric or some other yellow color, but the practice has now been largely discontinued. The pigments are readily seen in the siftings examined under a lens and their identity is established by simple micro-chemical tests.

<sup>&</sup>lt;sup>1</sup> U. S. Dept. Agr., Div. Chem., Bul. 13, p. 908.

<sup>&</sup>lt;sup>2</sup> Chemie der Menschlichen Nahrungs- und Genussmittel.

<sup>&</sup>lt;sup>8</sup> Canada Inland Revenue Dept., Bul. 24.

Facing of green tea with talc or clay and of black tea with plumbago and other black powders is also now seldom practiced.

Foreign Leaves and Spent Tea Leaves at one time were added to tea in the country of production. If a small handful of tea is brought to boiling with water and the leaves thus softened are spread out on paper, the form, size, and dentation of the leaves can be noted (Fig. 79). At the present time such an examination will seldom, if ever, disclose foreign leaves, but it will serve to bring out the size and maturity of the leaves, and the presence of stems and similar impurities.

The percentage of hot-water extract, as determined by a conventional method, was used to detect spent leaves.

Analysis of Tea. The methods described in Chapter IV are applicable to tea.

Caffeine is determined by direct weighing, as in the case of coffee, but the details of the process are different due to the presence of tannin and other interfering substances. One of the best processes is that of Stahlschmidt, as modified by Allen, in which the tannin is precipitated from a water infusion by lead acetate and the excess of lead in the filtered solution is removed by precipitation with sodium phosphate previous to extraction of the caffeine.

Tannin is estimated by oxidation with a standard solution of potassium permanganate, using indigo carmine as an indicator, as first proposed by Lowenthal and afterwards modified by Proctor.<sup>2</sup> Since other oxidizable substances are present it is necessary to make two titrations, one of the infusion directly to obtain the total oxidizable substances and another, after removal of the tannin by precipitation with gelatin. The difference between the two titrations represents the tannin.

<sup>&</sup>lt;sup>1</sup> Commercial Organic Analysis, 4th ed., Vol. VI, p. 607.

<sup>&</sup>lt;sup>2</sup> Jour. Soc. Chem. Ind., 3, p. 82.

#### CHOCOLATE AND COCOA

Composition of Cocoa Products. A summary of analyses of 17 varieties of unground *Chocolate* (Cocoa Nibs), made by Winton, Silverman, and Bailey 1 appears in the following table:

	Average.	Maximum.	Minimum
Water	2.72	3.18	2.29
Fat	50.12	52.25	48.11
Crude fiber	2.64	3.20	2.21
Theobromine	1.04	1.32	0.82
Caffeine	0.40	0.73	0.14
Protein	12.12	13.06	11.00
Ash	3.32	4.15	2.61
Starch	8.07	8.99	6.49
Nitrogen-free extract other than			
starch	19.57	21.07	17.69
	100.00		

COMPOSITION OF CHOCOLATE

The composition of *Cocoa* is the same as that of the chocolate from which it was made allowing for the fat removed and, in the case of so-called Dutch cocoa, for the alkali added to aid in forming a more complete emulsion in the preparation of the beverage.

Compounds of chocolate and cocoa with starch or flour are now unusual. Formerly they were sold fraudulently.

Cocoa Shells are used for preparing a mild beverage. When ground to an impalpable powder they are said to be added to cocoa.

Sweet Chocolate and Sweet Cocoa are mixtures containing sugar and often vanilla, vanillin, spices, or other flavors.

Milk Chocolate contains milk powder and usually also sugar and flavoring.

<sup>&</sup>lt;sup>1</sup> Conn. Agrl. Expt. Sta. Rept., 1902, p. 282.

Analysis of Chocolate and Cocoa. All the methods employed in the analysis of cereals and other natural vegetable foods, as described in Chapter IV, may be used for cocoa products. It should be noted, however, that the filtrations in the determination of fat and fiber are very slow and the method for starch requires preliminary extraction of the fat by ether or gasoline and of sugars (if present) by water.

Theobromine and Caffeine are determined by the Decker method.<sup>1</sup> The material, together with calcined magnesia, is boiled with water and the liquid filtered. The filtrate is evaporated to dryness and the residue extracted with chloroform. On evaporation of the chloroform a nearly pure mixture of the two alkaloids is obtained, which is weighed.

Caffeine is removed from the mixture by benzol, in which theobromine is insoluble at room temperature.

If a direct determination is desired the theobromine in the residue is treated according to Kunze's <sup>2</sup> method, based on the formation of silver theobromine when silver nitrate is added to an ammoniacal solution of the alkaloid.

<sup>&</sup>lt;sup>1</sup> Schweiz, Wchshr. Pharm., 1902, 40, pp. 527, 541, 553-

<sup>&</sup>lt;sup>2</sup> Ztschr. anal. Chemie, 1894, 33, p. 1.

## **APPENDIX**

## CALCULATION TABLES

# $\begin{array}{c} \textbf{VIETH'S TABLE FOR CORRECTING QUEVENNE LACTOMETER READ-} \\ \textbf{INGS FOR TEMPERATURE} \end{array}$

Degrees					D	egrees	of T	iermo	meter	(Fah	renhei	t),				
Lactom- eter.	45	46	47	48	49	50	51	52	53	54	55	56	57	58	59	60
											19.6					
21																
22																
											22.6					
											23.6					
25	23.8	23.9	24.0	24.0	24.1	24.1	24.2	24-3	24-4	24-5	24.6	24.6	24-7	24.8	24.9	-
26	2418	24.9	24.9	25.0	25.1	25.1	25.2	25.2	25-3	25-4	25-5	25.6	25-7	25.8	25.9	-
27																
28	26.7	26.8	26.8	26.9	27.0	27.0	27.1	27-2	27-3	27.4	27-5	27.6	27-7	27.8	27.9	-
29	27-7	27.8	27.8	27.9	28.0	28.0	28.1	28.2	28.3	28.4	28.5	28.6	28.7	28.8	28.9	-
30	28.6	28.7	28.7	28.8	28.9	29.0	29.1	29.1	29.2	29.3	29.4	29.6	29.7	29.8	29.9	-
31																
32	30.4	30.5	30.5	30.6	30.7	30.9	31.0	31.1	31.2	31.3	31.4	31.5	31.6	31.7	31.9	-
33	31.3	31.4	31.4	31.5	31.6	31.8	31.9	32.0	32.1	32-3	32.4	32.5	32.6	32:7	32.9	_
34	32.2	32.3	32.3	34-4	32.5	32-7	32.9	33.0	33.I	33-2	33-3	33-5	33.6	33-7	33-9	-
35	33.0	33.I	33.2	33-4	33-5	33-6	33.8	33-9	34.0	34-2	34-3	34-5	34.6	34-7	34.9	-
		61 ·	62	63	64	65	66	67	68	69	70	71	72	73	74	75
20																
21																
22																
23																
24		24.I	24.2	24.3	24-4	24.5	24.6	24-7	24.9	25.0	25.1	25.2	25-3	25.5	25.6	25-
25		25.I	25.2	25.3	25-4	25-5	25.6	25-7	25.9	26.0	26.1	26.2	26.4	26.5	26.6	26.1
20		20. I	26.2	20.3	26.5	26.6	20.7	20.8	27.0	27.1	27.2	27-3	27-4	27.5	27-7	27.
27																
28		28.1	28.3	28.4	28.5	28.6	28.7	28.8	29.0	29.1	29.2	29.4	29-5	29.7	29.8	29.9
29		29.1	29.3	29.4	29.5	29.6	29:8	29.9	30.1	30.2	30.3	30-4	30.5	30.7	30.9	31.0
30		30.1	30.3	30.4	30.5	30.7	30.8	30.9	31.1	31.2	31.3	31.5	31.6	31.8	31.9	32.1
31																
32		32.2	32.3	32.5	32.6	32.7	32.9	33.0	33.2	33-3	33-4	33.6	33-7	33-9	34.0	34-1
33		33.2	33-3	33-5	33.6	33.8	33-9	34.0	34.2	34-3	34.5	34.6	34-7	34.9	35.1	35 - 2
33 34		34.2	34-3	34-5	34.6	34.8	34.9	35.0	35.2	35-3	35-5	35.6	35.8	36 0	36.1	36.5
35		35.2	35-3	35-5	35.6	35.8	35.0	30. I	36.2	30.4	36.5	36.7	36.8	37.0	37.2	37.

LEACH'S TABLE FOR CALCULATING TOTAL SOLIDS IN MILK BY BABCOCK'S FORMULA FROM QUEVENNE LACTOMETER READING AND FAT.

Per			_			Lactor	neter l	Readin	g at 1	5.5° C.			-		
Cent f Fat.	22	23	24	25	26	27	28	20	30	31	32	33	34	35	36
0.0	5.50	5.75	6.00	6;25	6.50	6.75	7.00	7.25	7.50	7-75	8.00	8.25	8.50	8.75	9.0
0.1	5.62	5.87	6.12	6.40	6.62	6.87	7.12	7-37	7.62	7.87	8.12	8.37	8.62	8.87	9.1
0.3	5.74	6.11	6.36	6,61	6.86	7.11	7.36	7.01	7.86	8.11	8.36	8.61	8.86	9.11	9.3
0.4	5.08	6.23	6.48	6.73		7.23	7.48	7.73	7.98	8.23	8.48	8.73	8.99	9.23	9.4
0.5	6.10	6.35	6.72	6.97	7.10	7-35	7.72	7.07	8.22	8.47	8.72	8.97	9.10	9-35	9.6
0.7	6.34	6.50	6.84	7.00	7.34	75 50	7.72	7.97	8.34	8.50	8.84	9.00	9.34	9.50	9.8
0.8	6.46	6.83	7.08	7.21	7.46	7.71	7.96		8.46	8.71	8.00	9.21	9.46	9.71	9.9
1.0	6.70	6.95		7:45	7.70	New York	8,20	8 45	8.70	1200		No.		9.95	
I.I	6.82	7.07	7.52	7 - 57	.7.82	7.95	8.32	8.57	8.82	9.07	9.32	9.57	9.82	10.07	10.3
1.3	7.06	7.10	7-44	7.60	7.94	8.10	8.44	8.60	9.06		9.44	9.00	9.94	10.19	10.4
1.4	7.18		7.68	7.03	8.18	8.43	8.68	8.93	9.18	9.43	9.68	0.03	10.18	10.43	10.6
1.5	7.30	7.55	7.80	8.05	8.30	8.55	8.80	9.05	9.30		0.80	10.05	10.30	10.55	10.8
1.6	7.42	7.67	7.92	8.17	8.42	8.67	8.92		0.54	9.07	10.04	10.17	10.42	10.07	10.0
1.7	7.66	7.01	8.16	8.41	8.66	8.91	9.16	9.41	9.60	,0.01	10.16	10.41	10.66	10.91	II.I
1.9	7.78	8.03	8.28		100	9.03	9.28	9.53	9.78	10.03	10,28	10.55	10.78	11.04	11.2
3	7.00	8.15	8.40	8.65	8.00	9.15	9.40	9:65	0.00	10.15	10.40	10.66	10.91	11.16	11.4
2	8.14	8.30	8.64	8.89	9.14	9.30	0.64	9.89	10.14	10.27	10.64	10.95	11.15	11.40	11.
3	8.26	8.51	8.76	9.01	9.26	9.51	9.76	10.01	10.26	10.51	10.76	11.02	11.27	11.52	11.
25	8.38	8.63	8.88	9.13	9.38	9.03	10.00	10.25	10.50	10.75	11.00	11.26	11.51	11.76	12.0
4 5	8.50	8.75	9.12	9.37	0.62	9.87	10.12	10.37	10.62	10.75	11.12	11.38	11.63	11.88	12.
2.7	8.74	8.99	9.24	9.49	9.74	9.99	10.24	10.40	10.74	10.99	11.24	11.50	11.75	12.00	12
2.0	8.98	9.11	9.36	9.73	9.98	10,23	10.48	10.73	10.98	11.23	11.49	11.74	11.99	12.24	12.
310	9.10	9+35	9.60	9.85	10.10	10.35	10.60	10.85	11.10	11.36	11.61	11.86	12.11	12.36	12.0
3.1	9.22	9.47	9.72	9.97	10,22	10.47	10.72	11.00	11.35	11.48	11.73	12.10	12.23	12.40	12.
3.3	0.46	0.71	0.06	10.21	10.40	10.71	10.00	11.22	II.47	II.72	II. 07	12,22	12.48	12.73	13.0
3.4	9.58	9.83	10.08	10.33	10.58	10.83	11.00	11.34	11.50	11.84	12.09	12.34	12.60	12.85	13.1
3.5	0.82	10.07	10.32	10.45	10.10	11.08	11.33	11.58	11.83	11.96	12.33	12.58	12.84	13.00	13.
3.7	9.04	10.20	10.44	10.79	1 24	11.20	11.45	11.70	11.95	12.20	12.45	12.70	12.96	13.21	13.
3.8	10.06	10.43	10.56	10.81	18	11.32	11.57	11.82	12.07	12.32	12.57	12.82	13.08	13.33	13.
4.0	10.30	10.55	10.80	11.	.30	11.56	11.81	12.06	12.31	12.56	12.81	13.06	13.32	13.57	13.8
4. I			10.92		.42	11.68	11.93	12.18	12.43	12.68	12.93	13.18	13.44	13.69	13.
4.2	10.54	10.79	11.04	II.	1.66	11.02	12.05	12.42	12.67	12.80	13.18	13.43	13.68	13.04	14.0
4.4	10.78	11.03	11.28	II.	1.78	12.04	12.20	12.54	12.70	13.04	13.30	13.55	13.80	14.06	74.
4.5	10.00	11.15	11.40	II.	1.00	12.16	12.41	12.00	12.01	13.16	13.42	13.07	13.02	14.18	IA.
4.6			11.65		2.15	12.40	12.05	12.00	13.15	13.40	13.66	13.91	14.10	14.42	14.0
4.8	11.27	11.52	11.77	12.0	12.27	12.52	12.77	13.02	13.27	13.52	13.78	14.03	14.28	14.54	14-7
			11.89							13.64	200			10000	
5.0 5.1	11.63	11.88	12.01	12.1	12.63	12.88	13.13	13.38	13.63	13.76	14.14	14.30	14.64	14.00	15.1
5.2	11.75	12.00	12.25	12.5	12.75	13.00	13.25	13.50	13.75	14.01	14.26	14.51	14.76	15.02	15.2
5.3	11.87	12.12	12.37	12.01	12.87	13.12	13.37	13.02	13.87	14.13	14.38	14.75	14.88	15.14	15.3
5.4	12.11	12.36	12.61	12.86	13.11	13.36	13.01	13.86	14.12	14.25	14.62	14.88	15.13	15.38	15.0
5.6	12.23	12.48	12.73	12.98	13.23	13.48	13-73	13.99	14.24	14.40 14.61	14.75	15.00	15.25	15.50	15.7
5.7	12.35	12.72	12.05	13.10	13.47	13.72	13.07	14.22	14.48	14.74	14.00	15.24	15.40	15.74	15.0
5.9	12.59	13.84	13.00	13.34	13.59	13.84	14.10	14.35	14.60	14.74	15.11	15.36	15.61	15.86	16.1
6.0	12 21	12.06	13.21	12.46	13.71	13.06	14.22	TA. 47	14.72	14.08	15.22	15.48	15.72	TE. 08	16.3

TABLES

6	-			Invert and S	Sugar ucrose.		Lactose.		Mal	ose.	6
Cuprous Oxide (CusO).	Copper (Cu).	Dextrose.	Invert Sugar.	o.4 Gram Total Sugar.	s Grams Total Sugar.	СиНъОп.	CuHaOu+4HaO.	CµH#Ou+H10.	СиНиОп.	CuH#Ou+Ho.	Curon Onida (Curo)
10 11 13 14	8.9 9.8 10.7 11.5 12.4	4.6 4.5 4.9 5.3 5.7	4.5 5.0 5.4 5.8 6.3	116 2.1 2.5 2.0 3.4		3,8 4.5 5.1 5.8 0.4	3.9 4.6 5.3 5.9 6.6	4.0 4.7 5.4 6.1 6.8	5.9 6.7 7.5 8.3 9.1	6.2 7.0 7.9 8.7 9.5	1 1 1 1
15 16 17 18 19	13:3 14:2 15:1 16:0 16:9	6.2 6.6 7.0 7.5 7.9	7.2 7.6 8.1 8.5	3.2 4.3 4.8 5.2 5.7		7.1 7.8 8.4 9.1 9.7	7.3 8.0 8.6 9.3	7.5 8.2 8.9 9.5	79.9 10.6 11.4 12.2 13.0	10.4 11.2 12.0 12.9 13.7	1 1 1
20 21 22 23 24	17.8 18.7 19.5 20.4 21.3	8.3 8.7 9.2 9.6 10.0	8.9 9.4 9.8 10.3	6.1 6.6 7.0 7.5 7.9		io.4 II.0 II.7 I2.3 I3.0	10.7 11.3 12.0 12.7 13.4	10.9 11.6 12.3 13.0 13.7	13.8 14.6 15.4 16.2 17.0	14.6 15.4 16.2 17.1 17.9	2 2 1000
25 26 27 28 29	22.2 23.1 24.0 24.9 25.8	10,5 10.9 11.3 11.8	11.2 11.6 12.0 12.5 12.5	8.4 8.8 9.3 9.7 10.2		13.7 14.3 15.0 15.6 16.3	14.0 14.7 15.4 16.1 16.7	14.4 15.1 15.8 16.5 17.1	17.8 18.6 19.4 20.2 21.0	18.7 19.6 20.4 21.2 22.1	一次 元本の
30 31 32 33 34	26.6 27.5 28.4 29.3 30.2	12.6 13.1 13.5 13.9 14.3	13.4 13.8 14.3 14.7	10.7 11.1 11.6 12.0 12.5	4.3 4.7 5.2 5.6 6.1	16.9 17.6 18.3 18.9 19.6	17.4 18.1 18.7 19.4 20.1	17.8 18.5 19.2 19.9 20.6	21.8 22.6 23.3 24.1 24.9	22.9 23.7 24.6 25.4 26.2	3 3 3 3
35 36 37 38 39	31.1 32.0 32.9 33.8 34.6	14.8 15.2 15.6 16.1 16.5	15.6 16.1 16.5 16.9 17.4	12.9 13.4 13.8 14.3 14.7	6.5 7.0 7.4 7.9 8.4	20.2 20.9 21.5 22.2 22.8	20.8 21.4 22. 22. 23.	21.3 22.0 22.7 23.4 24.1	25.7 26.5 27.3 28.1 28.9	27.1 27.9 28.7 29.6 30.4	3 3 3 3 3
40 41 42 43 44	35.5 36.4 37.3 38.2 39.1	16.9 17.4 17.8 18.2 18.7	17.8 18.3 18.7 19.2 19.6	15.2 15.6 16.1 16.6 17.0	8.8 9.3 9.7 10.2 10.7	23.5 24.2 24.8 25.5 26.1	24. 24. 25.5 26.2	24.8 25.4 26.1 26.8 27.5	29.7 80.5 31.3 32.1 32.9	31.3 32.1 32.9 33.8 34.6	44444
45 46 47 48 49	40.0 40.9 41.7 42.6, 43.5	19.1 19.6 20.0 20.4 20.9	20.1 20.5 21.0 21.4 21.9	17.5 17.9 18.4 18.8 19.3	11.1 11.6 12.0 12.5 12.9	26.8 27.4 28.1 28.7 29.4	27.5 28.2 28.9 29.5 30-2	8.2 8.9 29.6 30.3 31.0	33.7 34.4 35.2 36.0 36.8	35.4 36.3 37.1 37.9 38.8	44444
50 51 52 53 54	44.4 45.3 46.2 47.1 48.0	21.3 21.7 22.2 22.6 23.0	22.3 22.8 23.2 23.7 24.1	19.7 20.2 20.7 21.1 21.6	13.4 13.9 14.3 14.8 15.2	30.1 30.7 31.4 32.1 32.7	30.9 31.5 32.2 32.9 33.6	31.7 32.4 33.0 33.7 34.4	37.6 38.4 39.2 40.0 40.8	39.6 40.4 41.3 42.1 42.9	5555
5% 56 57 58 59	48.9 49.7 50.6 51.5 52.4	23.5 23.9 24.3 24.8 25.2	24.6 25.0 25.5 25.9 26.4	22.5 22.5 22.9 23.4 23.9	15.7 16.2 16.6 17.1 17.5	33.4 34.0 34.7 35.4 26,0	34.3 34.9 35.6 36.3 37.0	35.1 35.8 36.5 37.2 37.9	41.6 42.4 43.2 44.0 44.8	43.8 44.6 45.4 46.3 47.1	5555
60 61 62 63 64	53.3 54.2 55.1 56.6 56.8	25.6 26.1 26.5 27.0 27.4	26.8 27.3 27.7 28.2 28.6	24.3 24.8 25.2 25.7 26.2	18.0 18.5 18.9 19.4 19.8	36.7 37.3 38.0 38.6 39.3	37.6 38.3 39.0 39.7 40.3	38.6 39.3 40.0 40.7 41.4	45.6 46.3 47.1 47.9 48.7	48.0 48.8 49.6 50.5 51.3	66666

### APPENDIX

## MUNSON AND WALKER'S TABLE FOR CALCULATING SUGARS FROM CUPROUS OXIDE—(Continued)

·(O;				Invert and Su	Sugar icrose.		Lactose.		Malt	ose.	(0)
Cuprous Oxide (CurO).	Copper (Cu).	Dextrose.	Invert Sugar.	o.4 Gram Total Sugar.	2 Grams Total Sugar.	СиНтОн.	CuHBOu+\$HiO.	CuH20u+H40.	CuHaOu.	Ci2H22O11 + H2O.	Cuprous Oxide (CusO.)
65	57.7	27.8	29.1	26.6	20.3	40.0	41.0	42.1	49.5	52.1	65
66	58.6	28.3	29.5	27.1	20.8	40.6	41.7	42.8	50.3	53.0	66
67	59.5	28.7	30.0	27.5	21.2	41.3	42.4	43.5	51.1	53.8	67
68	60.4	29.2	30.4	28.0	21.7	41.9	43.1	74.2	51.9	54.6	68
69	61.3	29.6	30.9	28.5	22.2	42.6	43.7	44.8	52.7	55.5	69
70	62.2	30.0	31.8	28.9	22,6	43.3	44.4	45.5	53.5	56.3	70
71	63.1	30.5	31.8	29.4	23.1	43.9	45.1	46.2	54.3	57.1	71
72	64.0	30.9	32.3	29.8	23.5	44.6	45.8	46.9	55.1	58.0	72
73	64.8	31.4	32.7	30.3	24.0	45.2	46.4	47.6	55.9	58.8	73
74	65:7	31.8	33.2	30.8	24.5	45.9	47.1	48.3	56.7	59.6	74
75	66.6	32.2	33.6	31.2	24.9	46.6	47.8	42.0	57.5	60.5	75
76	67.5	32.7	34.1	31.7	25.4	47.2	48.5	49.7	58.2	61.3	76
77	68.4	33.1	34.5	32.1	25.9	47.9	49.1	50.4	59.0	62.1	77
78	69.3	33.6	35.0	32.6	26.3	48.5	49.8	51.1	59.8	63.0	78
79	70.2	34.0	35.4	33.1	26.8	49.2	50.5	51.8	60.6	63.8	79
80 81 82 83 84	71.1 71.9 72.8 73.7 74.6	34.4 34.9 35.3 35.8 36.2	35.9 36.3 36.8 37.3 37.7	33.5 34.0 34.5 34.9	27.3 27.7 28.2 28.6 29.1	49.9 50.5 51.2 51.8 52.5	51.2 51.9 52.5 53.2 53.9	52.5 53.2 53.9 54.6 55.3	61.4 62.2 63.0 63.8 64.6	64.6 65.5 66.3 67.1 68.0	80 81 82 83 84
85	75.5.	36.7	38.2	35.8	29.6	53.1	54.6	56.0	65.4	68.8	85
86	76.4.	37.1	38.6	36.3	30.0	53.8	55.2	56.6	66.2	69.7	86
87	77.3	37.5	39.1	36.8	30.5	54.5	55.9	57.3	67.0	70.5	87
88	78.2	38.0	39.5	37.2	31.0	55.1	56.6	58.0	67.8	71.3	88
89	79.1	38.4	40.0	37.7	31.4	55.8	57.3	58.7	68.5	72.2	89
90	79.9	38.9	40.4	38.2	31.9	56.4	58.0	59.4	69.3	73.0	90
91	80.8	39.3	40.9	38.6	32.4	57.1	58.6	60.1	70.1	73.8	91
92	81.7	39.8	41.4	39.1	32.8	57.8	59.3	60.8	70.9	74.7	92
93	82.6	40.2	41.8	39.6	33.3	58.4	60.0	61.5	71.7	75.5	93
94	83.5	40.6	42.3	40.0	33.8	59.1	60.7	62.2	72.5	76.3	94
95	84.4	41.1	42.7	40.5	34.2	59.7	61:3	62.9	73.3	77.2	95
96	85.3	41.5	43.2	41.0	34.7	60.4	62.0	63.6	74.1	78.0	96
97	86.2	42.0	43.7	41.4	35.2	61.1	62.7	64.3	74.9	78.8	97
98	87.1	42.4	44.1	41.9	35.6	61.7	63.4	65.0	75.7	79.7	98
99	87.9	42.9	44.6	42.3	36.1	62.4	64.0	65.7	76.5	80.5	99
100 101 102 103 104	88.8 89.7 90.6 91.5 92.4	43.3 43.8 44.2 44.7 45.1	45.0 45.5 46.0 46.4 46.9	42.8 43.3 43.8 44.2 44.7	36.6 37.0 37.5 38.0 38.5	63.0 63.7 64.4 65.0 65.7	64.7 65.4 66.1 66.7 67.4	66.4 67.1 67.8 68.5 69.1	77.3 78.1 78.8 79.6 80.4	81.3 82.2 83.0 83.8 84.7	100 101 102 103 104
105	93.3	45.5	47.3	45.2	38.9	66.4	68.1	69.8	81.2	85.5	105
106	94.2	46.0	47.8	45.6	39.4	67.0	68.8	70.5	82.0	86.3	106
107	95.0	46.4	48.3	46.1	39.9	67.7	69.5	71.2	82.8	87.2	107
108	95.9	46.9	48.7	46.6	40.3	68.3	70.1	71.9	83.6	88.0	108
109	96.8	47.3	49.2	47.0	40.8	69.0	70.8	72.6	84.4	88.8	109
110	97.7	47.8	49.6	47.5	41:7	69.7	71.5	73.3	85.2	89.7	110
111	98.6	48.2.	50.1	48.0	41:7	70.3	72.2	74.0	86.0	90.5	111
112	99.5	48.7	50.6	48.4	42:2	71.0	72.8	74.7	86.8	91.3	112
113	100.4	49.1	51.0	48.9	42:7	71.6	73.5	75.4	87.6	92.2	113
114	101.3	49.6	51.5	49.4	43:2	72.3	74.2	76.1	88.4	93.0	114
115	102.2	50.0	51.9	49.8	43.6	73.0	74.9	76.8	89.2	93.9	115
116	103.0	50.5	52.4	50.3	44.1	73.6	75.6	77.5	90.0	94.7	116
117	103.9	50.9	52.9	50.8	44.6	74.3	76.2	78.2	90.7	95.5	117
118	104.8	51.4	53.3	51.2	45.0	75.0	76.9	78.9	91.5	96.4	118
119	105.7	51.8	53.8	51.7	45.5	75.6	77.6	79.6	92.3	97.2	119

TABLES

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# MUNSON AND WALKER'S TABLE FOR CALCULATING SUGARS FROM CUPROUS OXIDE—(Continued)

0).				Invert	Sugar icrose.	1	Lactose.		Mal	tose.	.(0)
Cuprous Oxide (CuiO).	Copper (Cu).	Dextrose.	Invert Sugar.	o.4 Gram Total Sugar.	s Grams Total Sugar.	СыНяОц.	CuHnOu+ \$ H10.	CuH#Ou+H4O	CuH#Ou.	CuHnOu+Ho.	Cuprous Oxide (CusO),
120	106.6	52.3	. 54.3	52.2	46.0	76.3	78.3	80.3	93.1	98.0	120
121	107.5	52.7	54.7	52.7	46.5	76.9	79.0	81.0	93.9	98.9	121
122	108.4	53.2	55.2	53.1	46.9	77.6	79.6	81.7	94.7	99.7	122
123	109.3	53.6	55.7	53.6	47.4	78.3	80.3	82.4	95.5	100.5	123
124	110.1	54.1	56.1	54.1	47.9	78.9	81.0	83.1	96.3	101.4	124
125	111.0	54.5.	56.6	54.5	48.3	79.6	81.7	83.8	97.1	102,2	125
126	111.9	55.0	57.0	55.0	48.8	80.3	82.4	84.5	97.9	103.0	126
127	112.8	55.4	57.5	55.5	49.3	80.9	83.0	85.2	98.7	103.9	127
128	113.7	55.9	58.0	55.9	49.8	81.6	83.7	85.9	99.4	104.7	128
129	114.6	56.3	58.4	56.4	50.2	82.2	84.4	86.6	100.3	105.5	129
130	115.5	56.8	58.9	56.9	50.7	82.9	85.1	87.3	101.0	106.4	130
131	116.4	57.2	59.4	57.4	51.2	83.6	85.7	88.0	101.8	107.2	131
132	117.3	57.7	59.8	57.8	51.7	84.2	86.4	88.7	102.6	108.0	132
133	118.1	58.1	60.3	58.3	52.1	84.9	87.1	89.4	103.4	108.9	133
134	119.0	58.6	60.8	58.8	52.6	85.5	87.8	90.1	104.2	109.7	134
135	119.9	59.0	61.2	59.3	\$3.1	86.2	88.5	90.8	105.0	110.5	135
136	120.8	59.5	61.7	59.7	53.6	86.9	89.1	91.5	105.8	111.4	136
137	121.7	60.0	62.2	60.2	54.0	87.5	89.8	92.1	106.6	112.2	137
138	122.6	60.4	62.6	60.7	54.5	88.2	90.5	92.8	107.4	113.0	138
139	123.5	60.9	63.1	61.2	55.0	88.9	91.2	93.5	108.2	113.9	139
140 141 142 143 144	124.4 125.2 126.1 127.0 127.9	61.3 61.8 62.2 62.7 63.1	63.6 64.0 64.5 65.0 65.4	61.6 62.1 62.6 63.1 63.5	55.5 55.9 56.4 56.9 57.4	89.5 90.2 90.8 91.5 92.2	91.9 92.5 93.2 93.9 94.6	94.2 94.9 95.6 96.3 97.0	109.0 109.8 110.5 111.3	114.7 115.5 116.4 117.2 118.0	140 141 142 143 144
145	128.8	63.6	65.9	64.0	57.8	92.8	95.8	97.7	112.9	118.9	145
146	129.7	64.0	66.4	64.5	58.3	93.5	95.9	98.4	113.7	119.7	146
147	130.6	64.5	66.9	65.0	58.8	94.2	96.6	99.1	114.5	120.5	147
148	131.5	65.0	67.3	65.4	59.3	94.8	97.3	99.8	115.3	121.4	148
149	132.4	65.4	67.8	65.9	59.7	95.5	98.0	100.5	116.1	122.2	149
150	133.2	65.9	68.3	66.4	60.2	96.1	98.7	101.2	116.9	123.0	150
151	134.1	66.3	68.7	66.9	60.7	96.8	99.3	101.9	117.7	123.9	151
152	135.0	66.8	69.2	67.3	61.2	97.5	100.0	102.6	118.5	124.7	152
153	135.9	67.2	69.7	67.8	61.7	98.1	100.7	103.3	119.3	125.5	153
154	136.8	67.7	70.1	68.3	62.1	98.8	101.4	104.0	120.0	126.4	154
155	137.7	68.2	70.6	68.8	62.6	99.5	102.1	104.7	120.8	127.2	155
156	138.6	68.6	71.1	69.2	63.1	100.1	102.8	105.4	121.6	128.0	156
157	139.5	69.1	71.6	69.7	63.6	100.8	103.4	106.1	122.4	128.9	157
158	140.3	69.5	72.0	70.2	64.1	101.5	104.1	106.8	123.2	129.7	158
159	141.2	70.0	72.5	70.7	64.5	102.1	104.8	107.5	124.0	130.5	159
160 161 162 163 164	142.1 143.0 143.9 144.8 145.7	70.4 70.9 71.4 71.8 72.3	73.0 73.4 73.9 74.4 74.9	71.2 71.6 72.1 72.6 73.1	65.0 65.5 66.0 66.5 66.9	102.8 103.4 104.1 104.8 105.4	105.5 106.2 106.8 107.5 108.2	108.2 108.9 109.6 110.3	124:8 125.6 126.4 127.8 128.0	131.4 132.2 133.0 133.9 134.7	160 161 162 163 164
165 166 167 168 169	146.6 147.5 148.3 149.2 150.1	72.8 73.2 73.7 74.1 74.6	75.3 75.8 76.3 76.8 77.2	73.6 74.0 74.5 75.0 75.5	67.4 67.9 68.4 68.9 69.3	106.1 106.8 107.4 108.1 108.8	108.9 109.6 110.3 110.9	111.7 112.4 113.1 113.8 114.5	128.8 129.6 130.3 131.1 131.9	135.5 136.4 117.2 134.0 138.0	165 166 167 168 169
170	151.0	75.1	77-7	76.0	69.8	109.4	112.3	115.2	132.7	139.7	170
171	151.9	75.5	78.2	76.4	70.3	110.1	113.0	115.9	133.5	140.5	171
172	152.8	76.0	78.7	76.9	70.8	110.8	113.7	116.6	134.3	141.4	173
173	153.7	76.4	79-1	77.4	71.3	111.4	114.3	117.3	135.1	142.2	173
174	154.6	76.9	79.6	77.9	71.7	112.1	115.0	118.0	135.9	143.0	174

0).			1	Invert and Su	Sugar icrose.		Lactose.		Malt	tose.	0).
Cuprous Oxide (CusO).	Copper (Cu).	Dextrose.	Invert Sugar.	o.4 Gram Total Sugar.	2 Grams Total Sugar.	СиНяОн.	CuHaOu+H40.	CuH#Ou+H50.	CuHnOu.	CuH20u+H20.	Cuprous Oxide (CusO).
175 176 177 178 179	155.5 156.3 157.2 158.1 159.0	77.4 77.8 78.3 78.8 79.2	80.1 80.6 81.0 81.5 82.0	78.4 78.8 79.3 79.8 80.3	72.2 72.7 73.2 73.7 74.2	112.8 113.4 114.1 114.8 115.4	115.7 116.4 117.1 117.8 118.4	118.7 119.4 120.1 120.8 121.5	136.7 137.5 138.3 139.1 139.8	143.9 144.7 145.5 146.4 147.2	17: 17: 17: 17:
180 181 183 183 184	159.9 160.8 161.7 162.6 163.4	79-7 80.1 80.6 81.1 81.5	82.5 82.9 83.4 83.9 84.4	80.8 81.3 81.7 83.2 82.7	74.6 75.1 75.6 76.1 76.6	116.1 116.7 117.4 118.1 118.7	119.1 119.8- 120.5 121.2 121.8	122.2 122.9 123.6 124.3 125.0	140.6 141.4 142.2 143.0 143.8	148.0 148.9 149.7 150.5 151.4	18 18 18 18
185 186 187 188 189	164.3 165.2 166.1 167.0 167.9	82.0 82.5 82.9 83.4 83.9	84.9 85.3 85.8 86.3 86.8	83.2 83.7 84.2 84.6 85.1	77.1 77.6 78.0 78.5 79.0	119.4 120.1 120.7 121.4 122.1	122.5 123.2 123.9 124.6 125.3	125.7 126.4 127.1 127.8 128 5	144.6 145.4 140.3 147.0 147.8	152.2 153.0 153.9 154.7 155.5	18 18 18 18
190 191 192 193 194	168.8 169.7 170.5 171.4 172.3	84.3 84.8 85.3 85.7 86.2	87.2 87.7 88.2 88.7 89.2	85.6 86.1 86.6 87.1 87.6	79.5 80.0 80.5 81.0 81.4	122.7 123.4 124.1 124.7 125.4	125.9 126.6 127.3 128.0 128.7	129.2 129.9 130.6 131.3 132.0	148.6 149.3 150.1 150.9 151.7	156.4 157.2 158.0 158.9 159.7	19 19 19 19
195 196 197 198 199	173.2 174.1 175.0 175.9 176.8	86.7 87.1 87.6 88.1 88.5	89.6 90.1 90.6 91.1 91.6	88.0 88.5 89.0 89.5 90.0	81.9 82.4 82.9 83.4 83.9	126.1 126.7 127.4 128.1 128.7	129.4 130.0 130.7 131.4 132.1	132.7 133.4 134.1 134.8 135.5	152.5 153.3 154.1 154.9 155.7	160.5 161.4 162.2 163.0 163.9	19 19 19
200 201 202 203 204	177.7 178.5 179.4 180.3 181.2	89.0 89.5 89.9 90.4 90.9	92.0 92.5 93.0 93.5 94.0	90.5 91.0 91.4 91.9 92.4	84.4 84.8 85.3 85.8 86.3	129.4 130.0 130.7 131.4 132.0	132.8 133.5 134.1 134.8 135.5	136.2 136.9 137.6 138.3 139.0	156.5 157.3 158.1 158.8 159.6	164.7 165.5 166.4 167.2 168.0	20 20 20 20
205 206 207 208 209	182.1 183.0 183.9 184.8 185.6	91.4 91.8 92.3 92.8 93.2	94.5 94.9 95.4 95.9 96.4	92.9 93.4 93.9 94.4 94.9	86.8 87.3 87.8 88.3 88.8	132.7 133.4 134.0 134.7 135.4	136.2 136.9 137.6 138.3 138.9	139.7 140.4 141.1 141.8 142.5	160.4 161.2 162.0 162.8 163.6	168.9 169.7 170.5 171.4 172.2	20 20 20 20 20
910 911 919 913 914	186.5 187.4 188.3 189.2 190.1	93.7 94.2 94.6 95.1 95.6	96.9 97.4 97.8 98.3 98.8	95.4 95.8 96.3 96.8 97.3	89.3 89.7 90.2 90.7 91.3	136.0 136.7 137.4 138.0 138.7	139.6 140.3 141.0 141.7 142.4	143.2 143.9 144.6 145.3 146.0	164.4 165.2 166.0 166.8 167.5	173.0 173.8 174.7 175.5 176.4	21 21 21 21
215 216 217 218 219	191.0 191.9 192.8 193.6 194.5	96.1 96.5 97.0 97.5 98.0	99:3 99.8 100.3 100.8 101.2	97.8 98.3 98.8 99.3 99.8	91.7 92.2 92.7 93.2 93.7	130.4 140.0 140.7 141.4 142.0	143.0 143.7 144.4 145.1 145.8	146.7 147.4 148.1 148.8 149.5	168.3 169.1 169.9 170.7 171.5	177.2 178.0 178.9 179.7 180.5	31 31 31
220 221 222 223 223	195.4 196.3 197.2 198.1	98.4 98.9 99.4 99.9 100.3	101.7 102.2 102.7 103.2 103.7	100.3 100.8 101.2 101.7 103.2	94.2 94.7 95.1 95.6 96.1	142.7 143.4 144.0 144.7 145.4	146.5 147.2 147.8 148.5 149.2	150.2 150.9 151.6 152.3 153.0	172.3 173.1 173.9 174.7 175.5	181.4 182.2 183.0 183.9 184.7	22 22 22 22
225 226 227 228 228	199 9 200.7 201.6 202.5 203.4	100.8 191.3 101.8 102.2 102.7	104.2 104.6 105.1 105.6 106.1	103.7 103.2 103.7 104.2 104.7	96.6 97.1 97.6 98.1 98.6	146.0 146.7 147.4 148.0 148.7	149.9 150.6 151.3 152.0 153.6	153.7 154.4 155.1 155.8 156.5	176.2 177.0 177.8 178.6 179.4	185.5 186.4 187.2 188.0 188.8	22 23 22 22 22

TABLES

(0)				Invert and St	Sugar	*	Lactose.		Mal	tose.	0
Cuprous Oxide (CutO)	Copper (Cu).	Dextrose.	Invert Sugar.	o.4 Gram Total Sugar.	2 Grams Total Sugar.	СиНяОп.	CuHmOu+4HsO.	CuHmOu+HiO.	СиНжОи.	CuHaOu+HaO.	Cuprous Oxide (CuiO)
230	204.3	103.2	106.6	105.2	99:1	149.4	153.3	157.2	180.2	189.7	23
231	205.2	103.7	107.1	105.7	99:6	150.0	154.0	157.9	181.0	190.5	23
232	206.1	104.1	107.6	106.2	100:1	150.7	154.7	158.6	181.8	191.3	23
233	207.0	104.6	108.1	106.7	100:6	151.4	155.4	159.3	182.6	192.2	23
234	207.9	105.1	108.6	107.2	101:1	152.0	156.1	160.0	183.4	193.0	23
235	208.7	105.6	109.1	107.7	101.0	152.7	156.7	160.7	184.2	193.8	23
236	209.6	106.0	109.5	108.2	102.1	153.4	157.4	161.4	184.9	194.7	23
237	210.5	106.5	110.0	108.7	102.6	154.0	158.1	162.1	185.7	195.5	23
238	211.4	107.0	110.5	109.2	103.1	154.7	158.8	162.8	186.5	196.3	23
239	212.3	107.5	111.0	109.6	103.5	155.4	159.5	163.5	187.3	197.2	23
240	213.2	108.0	111.5	110.1	104.0	156.1	160.2	164.3	188.1	198.0	24
241	214.1	108.4	112.0	110.6	104.5	156.7	160.9	165.0	188.9	198.8	24
242	215.0	108.9	112.5	111.1	105.0	157.4	161.5	165.7	189.7	199.7	24
243	215.8	109.4	113.0	111.6	105.5	158.1	162.2	166.4	190.5	200.5	24
244	216.7	109.9	113.5	112.1	106.0	158.7	162.9	167.1	191.3	201.3	24
245	217.6	110.4	114.0	112.6	106.5	159.4	163.6	167.8	192.1	202.2	24
246	218.5	110.8	114.5	113.1	107.0	160.1	164.3	168.5	192.9	203.0	24
247	219.4	111.3	115.0	113.6	107.5	160.7	165.0	169.2	193.6	203.8	24
248	220.3	111.8	115.4	114.1	108.0	161.4	165.7	169.9	194.4	204.7	24
249	221.2	112.3	115.9	114.6	108.5	162.1	166.3	170.6	195.2	205.5	24
250	222.1	112.8	116.4	115.1	109.0	162.7	167.0	171.3	196.0	206.3	25
251	223.0	113.2	116.9	115.6	109.5	163.4	167.7	172.0	196.8	207.2	25
252	223.8	113.7	117.4	116.1	110.0	164.1	168.4	172.7	197.6	208.0	25
253	224.7	114.2	117.9	116.6	110.5	164.7	169.1	173.4	198.4	208.8	25
254	225.6	114.7	118.4	117.1	111.0	165.4	169.8	174.1	199.2	209.7	25
255	226.5	115.2	118.9	117.6	111.5	166.1	170.5	174.8	200.0	210.5	25
256	227.4	115.7	119.4	118.1	112.0	166.8	171.1	175.5	200.8	211.3	25
257	228.3	116.1	119.9	118.6	112.5	167.4	171.8	176.2	201.6	212.2	25
258	229.2	116.6	120.4	119.1	113.0	168.1	172.5	176.9	202.3	213.0	25
259	230.1	117.1	120.9	119.6	113.5	168.8	173.2	177.6	203.1	213.8	25
260 261 262 263 264	231.8 231.8 232.7 233.6 234.5	117.6 118.1 118.6 119.0 119.5	121.4 121.9 122.4 122.9 123.4	120.1 120.6 121.1 121.6 122.1	114.5 115.0 115.5 116.0	169.4 170.1 170.8 171.4 172.1	173.9 174.6 175.3 176.0 176.6	178.3 179.0 179.8 180.5 181.2	203.9 204.7 205.5 206.3 207.1	214.7 215.5 216.3 217.2 218.0	26 26 26 26 26
265	235.4	120.0	123.9	122.6	116.5	172.8	177.3	181.9	207.9	218.8	26
266	236.3	120.5	124.4	123.1	117.0	173.5	178.0	182.6	208.7	219.7	26
267	237.2	121.0	124.9	123.6	117.5	174.1	178.7	183.3	209.5	220.5	26
268	238.1	121.5	125.4	124.1	118.0	174.8	179.4	184.0	210.3	221.3	26
269	238.9	122.0	125.9	124.6	118.5	175.5	180.1	184.7	211.0	222.1	26
270 271 272 273 274	239.8 240.7 241.6 242.5 243.4	122.5 122.9 123.4 123.9 124.4	126.4 126.9 127.4 127.9 128.4	125.1 125.6 126.2 126.7 127.2	119.5 120.0 120.6 121.1	176.1 176.8 177.5 178.1 178.8	180.8 181.5 182.1 182.8 183.5	185.4 186.1 186.8 187.5 188.2	211.8 212.6 213.4 214.2 215.0	223.0 223.8 224.6 225.5 226.3	27 27 27 27 27
275	244.3	124.9	128.9	127.7	121.6	179.5	184.2	188.9	215.8	227.1	27 27 27 27
276	245.2	125.4	129.4	128.2	122.1	180.2	184.0	189.6	216.6	228.0	
277	246.1	125.9	129.9	128.7	122.6	180.8	185.6	190.3	217.4	228.8	
278	246.9	126.4	130.4	129.2	123.1	181.5	186.3	191.0	218.2	229.6	
279	247.8	126.9	130.9	129.7	123.6	182.2	187.0	191.7	218.9	230.5	
280 281 282 283 284	248.7 249.6 250,5 251.4 252.3	127.3 127.8 128.3 128.8 129.3	131.4 131.9 132.4 132.9 133.4	130.2 130.7 131.2 131.7 132.2	124.1 124.6 125.1 125.6 126.1	182.8 183.5 ,184.2 184.8 185.5	187.7 188.3 189.0 189.7 190.4	192.4 193.1 193.9 194.6 195.3	219.7 220.5 221.3 222.1 222.9	231.3 232.1 233.0 233.8 234.6	28 28 28 28

6)				Invert and St	Sugar icrose.		Lactose.		Mal	tose.	(0)
Cuprous Oxide (CurO).	Copper (Cu).	Dextrose.	Invert Sugar.	9.4 Gram Total Sugar.	2 Grams Total Sugar.	СпНиОп.	Ctr H#O11 + # H2O	C11H#O11+H1O.	CuHmOu.	CnH#On+Ho.	Cuprous Oxide (CurO).
285 286 287 288 289	253.2 254.0 254.9 255.8 256.7	129.8 130.3 130.8 131.3 131.8	133.9 134.4 134.9 135.4 135.9	132.7 133.2 133.7 134.3 134.8	126.6 127.1 127.6 128.1 128.6	186.2 186.9 187.5 188.2 188.9	191.1 191.8 192.5 193.2 193.8	196.0 196.7 197.4 198.1 198.8	223.7 224.5 225.3 226.1 226.9	235.5 236.3 237.1 238.0 238.8	285 285 285 285
290 291 292 293 294	257.6 258.5 259.4 260.3 261.2	132.3 132.7 133.2 133.7 134.2	136.4 136.9 137.4 137.9 138.4	135.3 135.8 136.3 136.8 137.3	129.2 129.7 130.2 130.7 131.2	189 5 190 2 190.9 191.5 192 2	194.5 195.2 195.9 196.6 197.3	199 5 200 2 200 9 201 6 202 3	227.6 228.4 229.2 230 0 230.8	239.6 240-5 241.3 242.1 242.9	290 291 291 291 291
295 296 297 298 298	262.0 262.9 263.8 264.7 265.6	134.7 135.2 135.7 136.2 136.7	138 9 139 4 140.0 140.5 141.0	137.8 138.3 138.8 139.4 139.9	131.7 132.2 132.7 133.2 133.7	192 9 193 6 194 2 194 9 195 6	198.0 198.7 199.3 200.0 200.7	203.0 203.7 204.4 205.1 205.8	231 6 232.4 233 2 234 0 234.8	243.8 244.6 245.4 246.3 247.1	291 291 291 291 291
300 301 302 303 304	266.5 267.4 268.3 269.1 270.0	137.2 137.7 138.2 138.7 139.2	141.5 142.0 142.5 143.0 143.5	140.4 140.9 141.4 141.9 142.4	134.2 134.8 135.3 135.8 136.3	196 2 196.9 197.6 198 3 198 9	201.4 202.1 202.8 203.5 204.2	206.6 207.3 208.0 208.7 209.4	235.5 236.3 237.1 237.9 238.7	247.9 248.8 249.6 250.4 251.3	30 30 30 30
305 306 307 308 309	270.9 271.8 272.7 273.6 274.5	139.7 140.2 140.7 141.2 141.7	144.0 144.5 145.0 145.5 146.1	142:9 143:4 144:0 144:5 145:0	136.8 137.3 137.8 138.3 138.3	199.6 200 3 201 0 201.6 202 3	204.9 205.5 206.2 206.9 207.6	210.1 210 8 211.5 212.2 212.2	239.5 240.3 241.1 241.9 242.7	252.1 252.9 253.8 254.6 255.4	30 30 30 30
310 311 312 313 314	275.4 276.3 277.1 278.0 278.9	142.2 142.7 143.2 143.7 144.2	146.6 147.1 147.6 148.1 148.6	145.5 146.0 146.5 147.0 147.6	139.4 139.9 140.4 140.9 141.4	203.0 203.6 204.3 205.0 205.7	208.3 209.0 209.7 210.4 211.1	213 7 214.4 215 1 215.8 216.5	243.5 244.2 245.0 245.8 246.6	256.3 257.1 257.9 258.8 259.6	31: 31: 31: 31:
315 316 317 318 319	279.8 280.7 281.6 282.5 283.4	144.7 145.2 145.7 146.2 146.7	149.1 149.6 150.1 150.7 151.2	148.1 148.6 149.1 149.6 150.1	141.9 142.4 143.0 143.5 144.0	206.3 207.0 207.7 208.4 209.0	211.8 212.5 213.1 213.8 214.5	217 2 217 9 218 6 219 3 220 0	247.4 248.2 249.0 249.8 250.6	260 4 261 2 262 1 262 9 263 7	31 31 31 31
320 321 322 323 324	284.2 285 1 286 0 286.9 287 8	147.2 147.7 248.2 148.7 149.2	151.7 152.2 152.7 153.2 153.7	150.7 151.2 151.7 152.2 152.7	144.5 145.0 145.5 146.0 146.6	209.7 210.4 211.0 211.7 212.4	215.2 215.9 216.6 217.3 218 0	220.7 221.4 222.2 222.9 223 6	251.3 252.1 252:9 253.7 254.5	264 6 265 4 266 2 267 1 267 9	32 32 32 32 32 32
325 326 327 328 329	288 7 289.6 290.5 291.4 292.2	149.7 150.2 150.7 151.2 151.7	154.3 154.8 155.3 155.8 156.3	153 2 153.8 154.3 154.8 155.3	147.1 147.6 148.1 148.6 149.1	213.1 213.7 214.4 215.1 215.8	218.7 219 4 220.1 220.7 221.4	224.3 225 0 225 7 226 4 227 1	255 3 256 1 256.9 257 7 258.5	268.7 269 6 270 4 271.2 272.1	32 32 32 32 32
330 331 332 333 334	293.1 294.0 294.9 295.8 296.7	152.2 152.7 153.2 153.7 154.2	156.8 157.3 157.9 158 4 158 9	155 8 156.4 156.9 157.4 157.9	149.7 150.2 150.7 151.2 151.7	216 4 217.1 217.8 218.4 219.1	222.1 222 8 223.5 224.2 224.9	227.8 228 5 229.2 230.0 230.7	259.3 260 0 260 8 261 6 262 4	272.9 273.7 274.6 275.4 276.2	33: 33: 33: 33:
335 336 337 338 339	297.6 298.5 299.3 300.2 301 1	154.7 155.2 155.8 156.3 156.8	159.4 159.9 160.5 161.0	158.4 159.0 159.5 160.0	152.3 152.8 153.3 153.8 154.3	219.8 220.5 221.1 221.8 222.5	225.6 226.3 227.0 227.7 228.3	231.4 2 2.1 232.8 233.5 234.2	263 2 264.0 264.8 265.6 266.4	277.0 277.9 278 7 279.5 280.4	33 33 33 33

0).				Invert and Su	Sugar icrose.		Lactose.		Mal	tose.	(0)
Cuprous Oxide (Cud).	Copper (Cu).	Dextrose.	Invert Sugar.	o.4 Gram Total Sugar.	2 Grams Total Sugar.	CnHaOn.	CuH#Ou+\$H*O.	CuH#Ou+Ho.	CuH#Ôu.	CuHmOu+HrO.	Cuprous Oxide (CurO).
340	302.0	157.3	162.0	161.0	154.8	223.2	229.0	234.9	267.1	281.2	340
341	302.9	157.8	162.5	161.6	155.4	223.8	229.7	235.6	267.9	282.0	341
342	303.8	158.3	163.1	162.1	155.9	224.5	230.4	236.3	268.7	282.9	342
343	304.7	158.8	163.6	162.6	156.4	225.2	231.1	237.0	269.5	283.7	343
344	305.6	159.3	164.1	163.1	156.9	225.2	231.8	237.8	270.3	284.5	344
345	306.5	159.8	164.6	163.7	157.5	226.5	232.5	238.5	271.1	285.4	345
346	307.3	160.3	165.1	164.2	158.0	227.2	233.2	239.2	271.9	286.2	346
347	308.2	160.8	165.7	164.7	158.5	227.9	233.9	239.9	272.7	287.0	347
348	309.1	161.4	166.2	165.2	159.0	228.5	234.6	240.6	273.5	287.9	348
349	310.0	161.9	166.7	165.7	159.5	229.2	235.3	241.3	274.3	288.7	349
350	310.9	162.4	167.2	166.3	160.1	229.9	235.9	242.0	275.0	289.5	350
351	311.8	162.9	167.7	166.8	160.6	230.6	236.6	242.7	275.8	290.4	351
352	312.7	163.4	168.3	167.3	161.1	231.2	237.3	243.4	276.6	291.2	352
353	313.6	163.9	168.8	167.8	161.6	231.9	238.0	244.1	277.4	292.0	353
354	314.4	164.4	169.3	168.4	162.2	232.6	238.7	244.8	278.2	292.8	354
355	315.3	164.9	169.8	168.9	162.7	233.3	239.4	245.6	279.0	293.7	355
356	316.2	165.4	170.4	169.4	163.2	233.9	240.1	246.3	279.8	294.5	356
357	317.1	166.0	170.9	170.0	163.7	234.6	240.8	247.0	280.6	295.3	357
358	318.0	166.5	171.4	170.5	164.3	235.3	241.5	247.7	281.4	296.2	358
359	318.9	167.0	171.9	171.0	164.8	236.0	242.2	248.4	282.2	297.0	359
360	319.8	167.5	172.5	171.5	165.3	236.7	242.9	249.1	282.9	297.8	360
361	320.7	168.0	173.0	172.1	165.8	237.3	243.6	249.8	283.7	298.7	361
362	321.6	168.5	173.5	172.6	166.4	238.0	244.3	250.5	284.5	299.5	362
363	322.4	169.0	174.0	173.1	166.9	238.7	245.0	251.2	285.3	300.3	363
364	323.3	169.6	174.6	173.7	167.4	239.4	245.7	252.0	286.1	301.2	364
365	324.2	170.1	175.1	174.2	167.9	240.0	246.4	252.7	286.9	302.0	365
366	325.1	170.6	175.6	174.7	168.5	240.7	247.0	253.4	287.7	302.8	366
367	326.0	171.1	176.1	175.2	169.0	241.4	247.7	254.1	288.5	303.6	367
368	326.9	171.6	176.7	175.8	169.5	242.1	248.4	254.8	289.3	304.5	368
369	327.8	172.1	177.2	176.3	170.0	242.7	249.1	255.5	290.0	305.3	369
370	328.7	172.7	177.7	176.8	170.6	243.4	249.8	256.2	290.8	306.1	370
371	329.5	173.2	178.3	177.4	171.1	244.1	250.5	256.9	291.6	307.0	371
372	330.4	173.7	178.8	177.9	171.6	244.8	251.2	257.7	292.4	307.8	372
373	331.3	174.2	179.3	178.4	172.2	245.4	251.9	258.4	293.2	308.6	373
374	332.2	174.7	179.8	179.0	172.7	246.1	252.6	259.1	294.0	309.5	374
375	333.I	175.3	180.4	179.5	173.2	246.8	253.3	259.8	294.8	310.3	375
376	334.0	175.8	180.9	180.0	173.7	247.5	254.0	260.5	295.6	311.1	376
377	334.9	176.3	181.4	180.6	174.3	248.1	254.7	261.2	296.4	312.0	377
378	335.8	176.8	182.0	181.1	174.8	248.8	255.4	261.9	297.2	312.8	378
379	336.7	177.3	182.5	181.6	175.3	249.5	256.1	262.6	297.9	313.6	379
380	337.5	177.9	183.0	182.1	175.9	250.2	256.8	263.4	298.7	314.5	380
381	338.4	178.4	183.6	182.7	176.4	250.8	257.5	264.1	299.5	315.3	381
382	339.3	178.9	184.1	183.2	176.9	251.5	258.1	264.8	300.3	316.1	382
383	340.2	179.4	184.6	183.8	177.5	252.2	258.8	265.5	301.1	316.9	383
384	341.1	180.0	185.2	184.3	178.0	252.9	259.5	266.2	301.9	317.8	384
385	342.0	180.5	185.7	184.8	178.5	253.6	260,2	266.9	302.7	318.6	385
386	342.9	181.0	186.2	185.4	179.1	254.2	260.9	267.6	303.5	319.4	386
387	343.8	181.5	186.8	185.9	179.6	254.9	261.6	268.3	304.2	320.3	387
388	344.6	182.0	187.3	186.4	180.1	255.6	262.3	269.0	305.0	321.1	388
389	345.5	182.6	187.8	187.0	180.6	250.3	263.0	269.8	305.8	321.9	389
390	346.4	183.1	188.4	187.5	181.2	256.9	263.7	270.5	306.6	322.8	390
391	347.3	183.6	188.9	188.0	181.7	257.6	264.4	271.2	307.4	323:6	391
392	348.2	184.1	189.4	188.6	182.3	258.3	265.1	271.9	308.2	324.4	392
393	349.1	184.7	190.0	189.1	182.8	259.0	265.8	272.6	309.0	325.2	393
394	350.0	185.2	190.5	189.7	183.3	259.6	266.5	273.3	309.8	326.1	394

### APPENDIX

## MUNSON AND WALKER'S TABLE FOR CALCULATING SUGARS FROM CUPROUS OXIDE—(Continued)

0).				Invert	Sugar icrose.		Lactose.		Mal	tose.	6
Cuprous Oxide (CurO).	Copper (Cu).	Dextrose.	Invert Sugar.	o.4 Gram Total Sugar.	2 Grams Total Sugar.	СиНжОп.	CuH#Ou+ 1460.	CuHmOu+HiO.	СиНжОп.	C#H#On+H4O.	Cuprous Oxide (CusO),
395	350,9	185.7	191.0	190.2	183.9	260.3	267.2	274.0	310.6	326.9	395
396	351.8	186.2	191.6	190.7	184.4	261.0	267.9	274.7	311.4	327.7	396
397	352.6	186.8	192.1	191.3	184.9	261.7	268.6	275.5	312.1	328.6	397
398	353.5	187.3	192.7	191.8	185.5	262.3	269.3	276.2	312.9	329.4	398
399	354.4	187.8	193.2	192.3	186.0	263.0	269.9	276.9	313.7	330.2	399
400	335.3	188.4	193.7	192.9	186.5	263.7	270.6	277.6	314.5	331.1	400
401	356.2	188.9	194.3	193.4	187.1	264.4	271.3	278.3	315.3	331.9	401
402	357.1	189.4	194.8	194.0	187.6	265.0	272.0	279.0	316.1	332.7	402
403	358.0	189.9	195.4	194.5	188.1	265.7	272.7	279.7	316.9	333.6	403
404	358.9	190.5	195.9	195.0	188.7	266.4	273.4	280.4	317.7	334.4	404
405	359.7	191.0	196.4	195.6	189.2	267.1	274.1	281.1	318.5	335.2	405
406	360.6	191.5	197.9	196.1	189.8	267.8	274.8	281.9	319.2	336.0	406
407	361.5	192.1	197.5	196.7	190.3	268.4	275.5	282.6	320.0	336.9	407
408	362.4	192.6	198.1	197.2	190.8	269.1	276.2	283.3	320.8	337.7	408
409	363.3	193.1	198.6	197.7	191.4	269.8	276.9	284.0	321.6	338.5	409
410	364.2	193.7	199.1	198.3	191.9	270.5	277.6	284.7	322.4	339.4	410
411	365.1	194.2	199.7	198.8	192.5	271.2	278.3	285.4	323.2	340.2	411
412	366.0	194.7	200.2	199.4	193.0	271.8	279.0	286.2	324.0	341.0	412
413	366.9	195.2	200.8	199.9	193.5	272.5	279.7	286.9	324.8	341.9	-413
414	367.7	195.8	201.3	200.5	194.1	273.2	280.4	287.6	325.6	342.7	414
415	368.6	196.3	201.8	201.0	194.6	273.9	281.1	288.3	326.3	343.5	415
416	369.5	196.8	202.4	201.6	195.2	274.6	281.8	289.0	327.1	344.4	416
417	370.4	197.4	202.9	202.1	195.7	275.2	282.5	289.7	327.9	345.2	417
418	371.3	197.9	203.5	202.6	196.2	275.9	283.2	290.4	328.7	346.0	418
419	372.2	198.4	204.0	203.2	196.8	276.6	283.9	291.2	329.5	346.8	419
420	373.1	199.0	204.6	203.7	197.3	277.3	284.6	291.9	330.3	347-7	420
421	374.0	199.5	205.1	204.3	197.9	277.9	285.3	292.6	331.1	348.5	421
422	374.8	200.1	205.7	204.8	198.4	278.6	286.0	293.3	331.9	349-3	422
423	375.7	200.6	206.2	205.4	198.9	279.3	286.7	294.0	332.7	350.2	423
424	376.6	201.1	206.7	205.9	199.5	280.0	287.4	294.7	333.4	351.0	424
425	377.5	201.7	207.3	206.5	200.0	280.7	288.1	295.4	334.2	351.8	425
426	378.4	202.2	207.8	207.0	200.6	281.3	288.8	296.2	335.0	352.7	426
427	379.3	202.8	208.4	207.6	201.1	282.0	289.4	296.9	335.8	353.5	427
428	380.2	203.3	208.9	208.1	201.7	282.7	290.1	297.6	336.6	354.3	428
429	381.1	203.8	209.5	208.7	202.2	283.4	290.8	298.3	337.4	355.1	429
430	382.0	204.4	210.0	209.2	202.7	284.1	291.5	299.0	338.2	356.0	430
431	382.8	204.9	210.6	209.8	203.3	284.7	292.2	299.7	339.0	356.8	431
432	383.7	205.5	211.1	210.3	203.8	285.4	292.9	300.5	339.7	557.6	432
433	384.6	206.0	211.7	210.9	204.4	286.1	293.6	301.2	340.5	358.5	433
434	385.5	206.5	212.2	211.4	204.9	286.8	294.3	301.9	341.3	359.3	434
435	386.4	207.1	212.8	212.0	205.5	287.5	295.0	302.6	342.1	360.1	435
436	387.3	207.6	213.3	212.5	206.0	288.1	295.7	303.3	342.9	361.0	436
437	388.2	208.2	213.9	213.1	206.6	288.8	296.4	304.0	343.7	361.8	437
438	389.1	208.7	214.4	213.6	207.1	289.5	297.1	304.7	344.5	362.6	438
439	390.0	209.2	215.0	214.2	207.7	290.2	297.8	305.5	345.3	363.4	439
440	390.8	209 8	215.5	214.7	208.2	290.9	298.5	306.2	346.1	364.3	440
441	391.7	210.3	216.1	215.3	208.8	291.5	299.2	306.9	346.8	365.1	441
442	392.6	210.9	216.6	215.8	209.3	292.2	299.9	307.6	347.6	365.9	442
443	393.5	211.4	217.2	216.4	209.9	292.9	306.6	308.3	348.4	366.8	443
444	394.4	212.0	217.8	216.9	210.4	293.6	301.3	309.0	349.2	567.6	444
445	395.3	212.5	218.3	217.5	211.0	294.2	302.0	309.7	350.0	368.4	445
446	396.2	213.1	218.9	218.0	211.5	294.9	302.7	310.5	350.8	369.3	446
447	397.1	213.6	219.4	218.6	212.1	295.6	303.4	311.2	351.6	370.1	447
448	397.9	214 1	220.0	219.1	212.6	296.3	304.1	311.0	352.4	370.9	448
449	398.8	214.7	220.5	219.7	213.2	297.0	304.8	312.6	353.2	371.7	449

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## MUNSON AND WALKER'S TABLE FOR CALCULATING SUGARS FROM CUPROUS OXIDE—(Continued)

6				Invert and Su	Sugar scrose.		Lactose.		Malt	io <b>s</b> c.	 6
Cuprous Oxide (CusO).	Copper (Cu).	Dextrose.	Invert Sugar.	o.4 Gram Total Sugar.	2 Grams Total Sugar,	CuH±On.	CaHaOu+1H5O.	CuHaOu+Ho.	СиНаОи	CulfigOn + H <sub>2</sub> O.	Caprous Oxide (CusO).
450	399 · 7	215.8	221.1	220.2	213.7	297.6	305.5	313.3	353.9	372.6	450
451	400 · 6	215.8	221.6	220.8	214.3	298.3	306.2	314.0	354.7	373.4	451
452	401 · 5	216.3	222.2	221.4	214.8	299.0	306.9	314.7	355.5	374.2	452
453	402 · 4	216.9	222.8	221.9	215.4	299.7	307.6	315.5	350.3	375.1	453
454	403 · 3	217.4	223.3	222.5	215.9	300.4	308.3	316.2	357.1	375.9	454
455	404.2	218.0	223.9	223.0	216.5	301.7	309.0	316.9	357.9	376.7	455
456	405.1	218.5	224.4	223.6	217.0	301.7	309.7	317.6	358.7	377.6	456
457	405.9	219.1	225.0	224.3	217.6	302.4	310.4	318.3	359.5	378.4	457
458	406.8	219.6	225.5	224.7	218.1	303.1	311.1	319.0	360.3	379.2	458
459	407.7	220.2	220.1	225.3	218.7	303.8	311.8	319.8	361.0	380.0	459
460 461 462 463 464	408.6 409.5 410.4 411.3 412.2	220.7 221.3 221.8 222.4 222.9	226.7 227.2 227.8 228.3 228.9	225.8 226.4 226.9 227.5 228.1	219.2 219.8 220.3 220.9	304.5 305.1 305.8 306.5 307.2	312.5 313.2 313.9 314.6 315.3	320.5 321.2 321.9 322.6 323.4	361.8 362.6 363.4 364.2 365.0	380.9 381.7 382.5 383.4 384.2	460 461 462 463 464
465	413.0	223.5	229.5	228.6	222.0	307.9	316.0	324.1	365.8	385.0	465
466	413.9	224.0	230.0	229.2	222.5	308.6	316.7	324.8	366.6	385.9	466
467	414.8	224.0	230.6	229.7	223.1	309.2	317.4	325.5	367.3	386.7	467
468	415.7	225.1	231.2	230.3	223.7	309.9	318.1	326.2	368.1	387.5	468
469	416.6	225.1	231.7	230.9	224.2	310.6	318.8	326.9	368.9	388.3	469
470	417.5	220.2	232.3	231.4	224.8	311.3	319.5	327.7	369.7	389.2	470
471	418.4	226.8	232.8	232.0	225.3	312.0	320.2	328.4	370.5	390.0	471
472	419.3	227.4	233.4	232.5	225.9	312/6	320.9	329.1	371.3	390.8	472
473	420.2	227.9	234.0	233.1	226.4	313.3	321.6	329.8	372.1	391.7	473
474	421.0	228 5	234.5	233.7	227.0	314.0	322.3	330.\$	372.9	392.5	474
475	421.9	229 0	235.1	234.2	227.6	314.7	323.0	331.3	373.7	393.3	475
476	422.8	219.6	235.7	234.8	228.1	315.4	323.7	332.0	374.4	394.2	476
477	423.7	230.1	236.2	235.4	228.7	316.1	324.4	332.7	375.2	395.0	477
478	424.6	230.7	236.8	235.9	229.2	316.7	325.1	333.4	376.0	395.8	478
479	425.5	231.3	237.4	236.5	229.8	317.4	325.8	334.1	376.8	396.6	479
480 481 482 483 484	426.4 427.3 428.1 429.0 429.9	231.8 232.4 232.9 233.5 234.1	237.9 238.5 239.1 239.6 240.2	237.1 237.6 238.2 238.8 239.3	230.3 230.9 231.5 232.0 232.6	318.8 319.5 320.1 320.8	326.5 327.2 327:9 328.6 329.3	334.8 335.6 336.3 337.0 337.7	377.6 378.4 379.2 380.0 380.7	397 · 5 398 · 3 399 · 1 400 · 0 400 · 8	480 481 482 483 484
485	430.8	234.6	240.8	239.9	235.2	321.5	330.0	338.4	381.5	401.6	485
486	431.7	235.2	241.4	240.5	233.7	322.2	30.7	339.1	382.3	402.4	486
487	432.6	235.7	241.9	241.0	234.3	322.9	331.4	339.9	383.1	403.3	487
488	433.5	266.3	242.5	241.6	234.8	323.6	332.1	340.6	385.9	404.1	488
489	434.4	236.9	243.1	242.2	235.4	324.2	332.8	341.3	384.7	404.9	489
490	435.3	237.4	243.6	242.7	236.0	324. 9	333.5	342.0	385.5	405.8	490

APPENDIX

# EQUIVALENTS OF INDICES OF REFRACTION AND BUTYRO-REFRACTOMETER READINGS

Refrac-	Fourth Decimal of nD.													
Index.	0	1	2	3	4	5	-6	. 7	8	9				
				S	CALE RE	EADINGS	3.							
1.422	0.0	0.1	0.2	0.4	0.5	0.6	0.7	1 0.9	1.0	T.T				
I.423	1.2	1.4	1.5	1.6	1.7	1.9	2.0	2.I	2.2	2.4				
I.424	2.5	2.6	2-7	2.8	3.0	3.1	3.2	3-3	3-5	3.6				
1.425	3-7	3.8	4.0	4.I	4.2	4.3	4-5	4.6	4-7	4.8				
1.426	5.0	5.1 6.4	5-2	5-4	5.5	5.6	5-7	5.9	6.0	Ú.I				
I.427		0.4	6.5	6.6		6.9	7.0 8.2	7.I 8.4	7.2 8.5	7-4				
1.428	7.5	7.6	7-7	7-9	8.0	8.1			8.5	8.6				
1.429	8.7	8.9	9.0	9.1	9.2	9-4	9-5	9.6	9.8	9.9				
1.430	10.0	10.1	10.3	10.4	10.5	10.6	10.7	10.9	11.0	II-I				
1.431	11.3	11.4	11.5	.11.6	11.8	11.9	12.0	12.2	12-3	12.4				
1.432	12.5	12.7	12.8	12.9	13.0	13.2	13-3	13-5	13.6	13-7				
1.433		14.0	14.1	14.2	14.4	14.5	14.6	14.7	14.9	15.0				
1.434	15.1	15.3	15.4	15-5	15.6	15.8	15.9	16.0	16.2	16.3				
1.435	16.4	10.6	16.7	16.8	17.0	17.1	17.2	17.4	17.5	17.6				
1.436	17.8	17.9	19-3		19.6	19.7	19.8	20.0	20.1	20.3				
I-437 I-438	20.4	20.5	20.6	19.5	20.0	21.1	21.2	21.3	21.4	21.6				
1-439	21.7	21.8	22.0	22.I	22.2	22.4	22.5	22.6	22.7	22.9				
I.440	23.0	23.2	23-3	23.4	23.5	23.7	23.8	23.0	24.I	24.2				
1.441	24-3	24.5	24.6	24-7	24.8	25.0	25.1	25.2	25-4	25-5				
1.442	25.6	25.8	25.9	26.I	26.2	26.3	26.5	26.6	26.7	26.9				
I-443	27.0	27.1	27-3	27.4	27-5	27-7	27.8	27.9	28.1	28.2				
I.444	28.3	28.5	28.6	28.7	28.9	29.0	29-2	29.3	29.4	29.6				
1.445	29-7	29.9	30.0	30.1	30.3	30.4	30.6	30.7	30.8	30.9				
1:446	31.1	31.2	31.4	31-5	31.6	31.8	31.9	32.1	32.2	32.3				
I.447	32.5	32.6	32.8	32.9	33.0	33-2	33-3	33.5	33.6	33-7				
1.448	33-9	34.0	34-2	34.3	34.4	34.6	34-7	34.9	35.0	35-I				
1.449	35-3	35-4	35.6	35-7	35.8	36.0	36.1	36.3	36.4	36.5				
1.450	36.7	36.8	37.0	37-1	37-2	37-4	37-5	37-7	37.8	37-9				
1.451	38.1	38.2	38.3	38.5	38.6	38.7	38.9	39.0	39-2	39-3				
1.452	39,5	39.6	39-7	39-9	40.0	40.1	40.3	40.4	40.6	40.7				
1.453	40.9	41.0	41.1	41-3	41.4	41.5	41.7	41.8	42.0	42.I				
1.454	42.3	42.4	42.5	42-7	42.8	43.0	43.I	43-3	43-4	43.6				
1.455	43-7	43.9	44.0	44.2	44-3	44-4	44.6	44-7	44-9	45.0				
1.456	45.2	45-3	45.5	45.6	45-7	45.9	46.0	46.2	46.3	46.4				
1.457	48.0	48.2	48.3	47.0.	47-2 48.6	47·3 48.8	47.5	49.I	49.2	47-9				
1.458	49.5	49.7	49.8	50.0	50.1	50.2	50.4	50.5	50.7	50.8				
1.460	51.0	51.1	51.3	51-4	51.6	51.7	51.0	52.0	52.2	52.3				
1.461	52.5	52-7	52.8	53.0	53-1	53-3	53-4	53.6	53-7	53.9				
1.402	54.0	54.2	54-3	54-5	54.6	54.8	55.0	55.1	55-3	55-4				
1.463	55.6	55-7	55.9	56.0	56.2	56.3	56.5	56.6	56.8	56.9				
1.464	57.1	57.3		57.6	57-7	57.9	58.0	58.2	58.3	58.5				
1.465	57.1	57.3 58.8 60.3	57-4 58.9	59.1		59.4	59-5	59-7	59.8	60.0				
1.466	60.2	60.3	60.5	60.6	59.2	60.9	61.1	61.2	61.4	61.5				
1,467	61.7	61.8	62.0	62.2	62.3	. 62.5	62.6	62.8	62.9	63.1				
1.468	63.2	63.4	63 5	63.7	63.8	64.0	64.2	64.3	64.5	64-7				
1.469	64.8	65.0	65.1	65.3	65.4	65.6	65-7	65.9	66.1	66.2				

TABLES

EQUIVALENTS OF INDICES OF REFRACTION AND BUTYRO-REFRACTOMETER READINGS—(Continued)

Refrac-	Fourth Decimal of $*_{D_*}$													
Index.	0	1	2	3	4	5	6	7	8	9				
				80	CALE RI	EADÍNGS	J.							
1.470	66.4	66.5	66.7	66.8	67.0	67.2	67.3	67.5	67.7	67.8				
1.471	68.0	68.i	68.3	68.4	68.6	68.7	68,6	69.I	69.2	69.4				
1.472	69.5	69.7	69.9	70.0	70.2	70.3	70.5	70.7	70.8	71.0				
1:473	71.1	71.3	71.4	71.6	71.8	71.9	72.1	72.2	72.4	72.5				
1.474	72.7	72.9	73.0	73.2	73-3	73-5	73-7	73.8	74.0	74-1				
1.475	74-3	74.5	74.6	74.8	75.0	75.1	75-3	75.5	75.6	75.8				
1.476	76.0	76.1	76.3	76.5	76.7	76.8	77.0	77-2	77-3	77-5				
1.477	77 - 7	77-9	78. I	78.2	78.4	78.6	78.7	78.9	79.I	79-2				
1.478	79-4	79.6	79.8	80.0	80. ī	80.3	80.5	80.6	80.8	81.0				
1-479	81.2	81.3	81.5	81.7	81.9	82.0	82.2	82.4	82.5	82.7				
1.480	82.9	83.1	83.2	83.4	83.6	83.8	83.9	84.1	84.3	84.5				
1.481	84.6	84.8	85.0	85.2	85.3	85.5	85.7	85.9	86.0	86.2				
1.482	86.4	86.6	8ő. <sub>7</sub>	8ŏ.9	87.1	87.3	87.5	87.6	87.8	88.0				
1.483	88.2	88.3	88.5	88.7	88.9	89.1	89.2	89.4	89.6	89.8				
1.484	90.0	90.2	90.3	90.5	90.7	90.9	91.1	91.2	91.4	91.6				
1.485	91.8	92.0	92.1	92.3	92.5	92.7	92.9	93.0	93-2	93.4				
1.486	93.6	93.8	94.0	94.1	94.3	94-5	94.7	94.8	95.0	95.2				
1.487	95-4	95.6	95.8	96.0	96.1	96.3	96.6	96.7	96.9	97.0				
1.488	97.2	97-4	97.6	97.8	98.o	98.1	98.3	98.5	98.7	98.9				
1.489	99.1	99.2	99.4	99.6	99.8	100.0				1				

#### GEERLIG'S TABLE FOR CALCULATING DRY SUBSTANCE OF SAC-CHARINE PRODUCTS FROM REFRACTIVE INDEX AT 28° C.

Find in the table the refractive index which is next lower than the reading actually made and note the corresponding whole number for the per cent of dry substance. Subtract the refractive index obtained from the table from the observed reading; the decimal corresponding to this difference, as given in the column so marked, is added to the whole per cent of dry substance as first obtained.

Refrac- tive Index.	Per Cent Dry Sub- stance.	Decimals to b Fractional		Refrac- tive Index.	Per Cent Dry Sub- stance.	Decimals to be Added for Fractional Readings.
1.3335	1	0.0001=0.05	0.0010=0.75	1.4083	45	0.0004-0.2 0.0015-0.75
1.3349	2	0.0002=0.1	0.0011=0.8	1.4104	46	0.0005=0.250.0016=0.8
1.3364	3	0.0003=0.2	0.2012=0.8	1.4124	47	0.0006=0.3 0.0017=0.85
1.3379	4	0.0004=0.25	0.0013=0.85	1.4145	48	0.0007=0.350.0018=0.0
1.3394		0.0005=0.3	0.0014=0.0	1.4166	49	0.0008=0.4 0.0010=0.05
1.3400	5 6	0.0006=0.4	0.0015=1.0	1.4186	50	0.0009=0.45 0.0020=1.0
1.3424	7 8	0.0007=0.5	•	1.4207	51	0.0010=0.5 0.0021=1.0
1.3439	8	0.0008=0.6		1.4228	52	0.0011=0.55
1.3454	9	0.0009=0.7		1.4219	53	- <del>-</del>
1.3469	10			1.4270	54	
1.3484	11	0.0007=0.05		1.4292	55	0.0001=0.05 0.0013=0.55
1.3500	12	0.0002=0.I		1.4314	56	0.0002=0.1 0.0014=0.6
1.3510	13	0.0003=0.2		1.4337	57	0.0003=0.1 0.0015=0.65
1.3530	14	0.0004=0.25		1.4359		0.0004=0.150.0016=0.7
1.3546	15	0.0005=0.3		I.4382	59 60	0.0005=0.2 0.0017=0.75
1.3578	17	0.0007=0.45		1.4428		0.0007=0.3 0.0019=0.85
1.3594	18	0.0008=0.5		1.4451		0.0008=0.350.0020=0.0
1.3611	19	0.0009=0.6		1.4474	63	0.0009=0.4 0.0021=0.0
1.3627	20	0.0010=0.6		1.4497		0.0010=0.450.0022=0.95
.3644	21	0.0011=0.7		1.4520		0.0011=0.5 0.0023=1.0
.3661	22	0.0012=0.75		1.4543	66	0.0012=0.5 0.0024=1.0
3678	23	0.0013=0.8		1.4567	67	,
1.3695	24	0 0014=0.85		1.4591	68	i l
1.3712	25	0.0015=0.4		1.4615	69	
1.3729	26	0.0016=0.		1.4639		
				1.4663		
1.3746	27	0.0001=0.5	0.0012=0.6		′-	
1.3764	28	0.0002=0.	0.0013=0.65			
1.3782	29	0.0003=0.5	0.0014=0.7	1.4711		0.0001=0.0 0.0015=0.55
1.3800	30	0.0004=0.2	0.0015=0.75	1.4736		0.0002=0.05 0.0016=0.6
1.3818	31	0.0005=0.25	0.0016=0.8 0.0017=0.85	1.4761		0.0003=0.1 0.0017=0.65
1.3836	32	0.0007=0.35	0.0017=0.05	1.4786		0.0005=0.2 0.0019=0.7
1.3872	34	0.0008=0.45		1.4836		0.0006=0.2 0.0020=0.75
1.3890	35	0.0009=0.4	0.0020=1.0	1.4862		0.0007=0.250.0021=0.8
1.3690	36	0.0010=0.5	0.0021=1.0	1.4888		0.0008=0.3 0.0022=0.8
1.3928	37	0.0011=0.55		1.4914		0.0009=0.350.0023=0.85
1.3947	38	]		1.4940	1 2	0.0010=0.350.0024=0.9
1.3966	39			1.4966		0.0011=0.4 0.0025=0.9
1.3984	40			1.4992		0.0012=0.45 0.0026=0.95
1.4003	41			1.5019		0.0013=0.5 0.0027=1.0
	I			1.5046	86	0 0014=0.5 0.0028=1.0
	1			1.5073		1
1.4023	42	0.0001=0.05		1.5100		1 1
1.4043	43	0.0002=0.1	0.0013=0.65		-	1
1.4063	44	0.0003=0.15	0.0014=0.7	1.5155	90	l d

TABLES

TEMPERATURE CORRECTIONS FOR USE WITH GEERLIG'S TABLE, PAGE 224

Tempera-	17	Dry Substance.														
ture of the	0	5	10	15	20	25	30	40	50	60	70	80	90			
°C.							Subtra	ct—								
20	0.53	0.54	0.55	0.56	0.57	0.58	0.60	0.62	0.64	0.62	0.61	0.60	0.58			
21	.46		.48	-49	-50	-51	-52	-54	-56	-54	-53	-52	-50			
22	.40	-41	-42	-42	-43	-44	-45	-47	.48	-47	.46	-45	-44			
23	-33	-33	-34	-35	.36	-37	-38	-39	.40	-39	-38	.38	.38			
24	.26	.26	.27	.28	. 28	-29	-30	-31	.32	-31	-31	-30	-30			
25	-20	.20	.21	.21,	.22	.22	-23	.23	.24	-23	-23	.23	.22			
25	.12	.12	.13	.14	.14	.15	.15	.16	.16	.16	.15	.15	-14			
27	.07	-07	.07	.07	.07	.07	.08	-08	.08	.08	.08	.08	-07			
							Add	_								
29	0.07	0.07	0.07	0.07	0.07	0.07	0.08	0.08	0.08	0.08	0.08	0.08	0.07			
30	.12	.12	.13	-14	.14	.14	.15	.15	-16	.16	.16	.15	.14			
31	20	.20	.21	.21	.22	.22	-23	-23	.24	.23	.23	.23	.22			
32	. 26	.26	.27	.28	. 28	.29	.30	-31	-32	-31	.31	-30	.30			
33	-33	-33	-34	-35	.36	-37	-38	-39	.40	-39	-38	-38	.38			
34	.40	-41	.42	.42	-43	-44	-45	-47	-48	-47	.46	-45	-44			
35	.46	-47	.48	-49	-50	.51	-52	-54	.56	-54	-53	-52	-50			

HEHNER'S TABLE FOR CALCULATING ALCOHOL FROM SPECIFIC GRAVITY

	Abso	lute Alc	ohol.		Abso	lute Ale	ohol.		Abso	lute Alc	ohol.
Spec. Grav. at 15.6° C.	Per Cent by Weight	Per Cent by Vol- ume.	Grams per 100 cc.	Spec. Grav. at 15.6° C.	Per Cent by Weight	Per Cent by Voi- ume.	Grams per 100 cc.	Spec. Grav. at 15.6° C.	Per Cent by Weight	Per Cent by Vol- ume.	Grams per- 100 cc
1.0000	0.00	0.00	0.00								
0.9999	0.05	0.07	0.05	0.9959	2.33	2.93	2.32	0.9919	4.69	5.86	4.65
8	0.11	0.13	0.11	8	2.39	3.00	2.38	8	4-75	5-94	4-71
7	0.16	0.20	0.16	7	2.44	3.07	2.43	7	4.81	6.02	4-77
6	0.21	0.26	0.21	6	2.50	3.14	2.49	6		6.10	4.83
5	0.26	0.33	0.26	5	2.56	3.21	2.55	5		6.17	4.90
4	0.32	0.40	0.32	4	2.61	3.28	2.60	4		6.24	4-95
3	0.37	0.46	0.37	3	2.67	3-35	2.65	3		6.32	5.01
2	0.42	0.53	0.42	2	2.72	3-42	2.70	. 2	3	6.40	5-07
1	0.47	0.60	0.47	1	2.78	3-49	2.76	0	33	6.48	5.14
0	0.53	0.66	0.53	0	2.83	3-55	2.01	0	5-25	6.55	5.20
0.9989	0.58	0.73	0.58	0.9949	2.89	3.62	2.87	0.9909	5-31	6.63	5.26
8	0.63	0.79	0.63	. 8	2.94	3.69	2.92	8	5.37	6.71	5-32
7 6	0.68	0.86	0.68	7	3.00	3-76	2.98	7	5-44	6.78	5-39
	0.74	0.93	0.74	6	3.06	3.83	3.04	6	5.50	6.86	5-45
5	0.79	0.99	0.79	5	3.12	3.90	3.10	5	5.56	6.94	5-5I
4	0.84	1.06	0.84	4	3.18	3.98	3.16	4		7.01	5.57
3	0.89	1.13	0.89	3	3-24	4.05	3.22	3	5.69	7.09	5.64
2	0.95	1.19	0.95	2	3.29	4.12	3.27	2		7.17	5.70
1	1.00	1.26	1.00	.1	3-35	4.20	3-33	1		7-25	5.76
0	1.06	1.34	1.06	0	3.41	4.27	3-39	0	5.87	7-32	5.81
0.9979	1.12	1.42	1.12	0.9939	3.47	4-34	3-45	0.9899	5-94	7-40	5.88
8	1.19	1.49	1.19	8	3-53	4.42	3-51	8	6.00	7-48	5.94
7 6	1.25	1.57	1.25	7	3.59	4-49	3-57	7	6.07	7-57	6.01
	1.31	1.65	1.31	6	3.65	4.56	3.63	6	The Control of the Co	7.66	6.07
5	1.37	1.73	1.37	5	3.71	4.63	3.69	5	6.21	7-74	6.14
4	1.44	1.81	1.44	4	3.76	4-71	3-74	4	6.28	7.83	6.21
3	1.50	1.88	1.50	3	3.82	4-78	3.80	3		7-92	6.29
2	1.56	1.96	1.56	2	3.88	4.85	3.85	.2	6.43	8.01	6.36
0	1.62	2.04	1.61	1 0	3.94	4-93	3.91	1 0	6.50	8.10	6.43
٥	1.69	2.12	1.68	0	4.00	5.00	3.97		6.57	8,18	6.50
0.9969	1.75	2.20	1-74	0.9929	4.06	5.08	4.03	0.9889	6.64	8-27	6.57
8	1.81	2.27	1.80	8	4.12	5.16	4.00	. 8	6.71	8.36	6.63
7 6	1.87	2-35	1.86	7	4.19	5-24	4.16	7	6.78	8.45	6.70
	1.94	2.43	1.93	6	4-25	5-32	4.22	6	6.86	8.54	6.78
5	2.00	2.51	1.99	5	4.31	5-39	4.28	5	6.93	8.63	6.85
4	2.06	2.58	2.05	4	4-37	5-47	4-34	4	7.00	8.72	6.92
3	2.11	2.62	2.10	3	4-44	5-55	4-40	3	7.07	8.88	6.99
2	2.17	2.72	2.16	2	4.50	5.63	4.46	2	7-13	8.96	7.05
0	2.28	2.79	2.21		4.56	5.71	4-52	0	7 20	9.04	7.12
0	2.20	4.00	2.2/	9	4.02	2.10	4-58	0	11	9.04	1.29

HEHNER'S TABLE FOR CALCULATING ALCOHOL FROM SPECIFIC GRAVITY—(Continued)

7. C. 9879 7. 6. 7. 7. 6. 7. 7. 7. 7. 7. 7. 7. 7. 7. 7. 7. 7. 7.	33 9.1; 40 9.2; 47 9.2; 47 9.2; 60 9.4; 67 9.5; 67 9.7; 93 9.8; 90 9.9; 91 10.1; 10.1; 21 10.2; 21 10.3; 36 10.3;	3 7-24 7-31 7-37 7-43 7-57 7-53 7-57 2 7-63 7-70 3 7-70 7-83 7-83 7-96 8 8-94 8 8-10	5pc. Grav. at 15.6° C. 0.9829 8 7 6 5 4 3 .2 1 1 5 0 0 0 0 0 0 0 0 0 0 0 0 0 0 0 0 0	11.46	13.52 13.62 13.71 13.81 13.90 13.99 14.09 14.18 14.27 14.37	Grams per 10-73 10-81 10-89 11-93 11-11 11-18 11-26 11-31 11-41	Spec. Grav. at 15.6° C. O-9779 8 7 6 5 4 3 2 2 1 0 0	15.50 15.58 15.67	18.68 18.78 18.88 18.98 19.08	Grams per 100 cc.  14.58 14.66 14.74 14.83 14.90 14.98 15.07 15.14 15.21
8 7. 7 7. 6 7. 4 7. 3 7. 2 7. 0 7. 0 .9869 8. 8 8. 6 8.	47 9.24 47 9.24 47 9.24 553 9.33 660 9.44 67 9.56 73 9.66 80 9.77 87 9.71 93 9.86 00 9.99 00 10.00 114 10.11 221 10.22 29 10.33 36 10.33	7.31 7.37 7.43 7.50 7.50 7.50 7.63 7.70 7.83 7.89 7.89 7.80 8.04 8.10	8 7 6 5 4 3 2 1 0 0 0.9819 8	11.00 11.08 11.15 11.23 11.31 11.38 11.46 11.54 11.69	13.62 13.71 13.81 13.90 13.99 14.09 14.18 14.27 14.37	10.81 10.89 10.95 11.03 11.11 11.18 11.26 11.33 11.41	8 7 6 5 4 3 2	15.00 15.08 15.17 15.25 15.33 15.42 15.50 15.58 15.67	18.48 18.58 18.68 18.78 18.88 18.98 19.08	14.66 14.74 14.83 14.90 14.98 15.07 15.14 15.21 15.30
7 7. 6 7. 5 7. 3 7. 2 7. 7 7. 0 7. 0 8. 8 8. 6 8.	47 9.26 53 9.37 660 9.44 667 9.56 73 9.66 80 9.77 93 9.67 90 9.97 10.07 10.01 10.12 21 10.22 229 10.33 36 10.33	7-37 7-43 7-50 7-57 7-50 7-57 7-63 7-77 7-83 7-89 7-89 8-96 8-96 8-94 8-10	76 55 4 3.2 11 0	11.08 11.15 11.23 11.31 11.38 11.46 11.54 11.69	13.71 13.81 13.90 13.99 14.09 14.18 14.27 14.37	10.89 10.95 11.03 11.11 11.18 11.26 11.33 11.41	7 6 5 4 3 2	15.08 15.17 15.25 15.33 15.42 15.50 15.58 15.67	18.58 18.68 18.78 18.88 18.98 19.08	14-74 14-83 14-90 14-98 15-07 15-14 15-21 15-30
6 7. 5 7. 4 7. 3 7. 2 7. 1 7. 0 7. 0.9869 8. 8 8. 7 8. 6 8.	53 9-3 60 9-4 67 9-5 67 9-5 73 9-6 80 9-7 87 9-7 93 9-8 00 9-9 00 9-9 10 0-1 10 10 10 10 10 10 10 10 10 10 10 10 10 1	7. 43 7.50 7.57 7.63 7.70 7.77 7.83 7.89 7.96 8.04 8.10	6 5 4 3 2 1 0	11.15 11.23 11.31 11.38 11.46 11.54 11.62	13.81 13.90 13.99 14.09 14.18 14.27 14.37	10.95 11.03 11.11 11.18 11.26 11.33 11.41	6 5 4 3 2	15.17 15.25 15.33 15.42 15.50 15.58 15.67	18.68 18.78 18.88 18.98 19.08	14.83 14.90 14.98 15.07 15.14 15.21 15.30
4 7- 3 7- 2 7- 1 7- 0 7- 0 -9869 8- 8 8- 7 8- 6 8- 5 8- 8 8-	67 9.5. 73 9.6. 80 9.7. 87 9.7. 93 9.8. 00 9.9. 00 10.0. 14 10.1: 21 10.2: 22 10.3. 36 10.3.	7.57 7.63 7.79 7.77 7.83 7.89 7.96 8.04 8.10	0.9819	11.31 11.38 11.46 11.54 11.62	13.99 14.09 14.18 14.27 14.37	11.11 11.18 11.26 11.33 11.41	4 3 2 1	15.33 15.42 15.50 15.58 15.67	18.88 18.98 19.08 19.18	14.98 15.07 15.14 15.21 15.30
3 7- 2 7- 1 7- 0 7- 0.9869 8- 8 8- 7 8- 6 8- 5 8-	73 9.6: 80 9.7: 87 9.7: 93 9.8: 00 9.9: 07 10.0: 14 10.1: 21 10.2: 29 10.3: 36 10.3:	7.63 7.79 7.77 7.83 7.89 7.96 8.04 8.10	0.9819	11.38 11.46 11.54 11.62	14.09 14.18 14.27 14.37	11.18 11.26 11.33 11.41	3 2 1	15.42 15.50 15.58 15.67	19.08	15.14 15.21 15.30
7. 0.9869 8. 8 8. 7 8. 6 8. 5 8.	87 9-76 93 9-86 00 9-93 07 10-03 14 10-13 21 10-23 29 10-36 36 10-38	7-77 7-83 7-89 7-96 8-04 8-10	0.9819	11.62	14-27	11.33	1	15.58	19.18	15.21
0.9869 8. 8 8. 7 8. 6 8. 5 8.	93 9.86 00 9.93 07 10.03 14 10.13 21 10.23 29 10.36 10.38	7.83 7.89 7.96 8.04 8.10	0.9819	11.69			0	15.67		
8 8. 7 8. 6 8. 5 8.	07 10.0 14 10.1 21 10.2 29 10.3 36 10.3	7.96 8.04 8.10	8		14.46	0	100			2000
7 8. 6 8. 5 8.	14 10.13 21 10.2 29 10.30 36 10.30	8.04		11.77		11.48	0.9769			
6 8.	21 10.2 29 10.3 36 10.3	8.10		11.85		11.56	8 7	15.83		15.46
	36 10.3	N 2 74	6	11.92	14-74	11.70	6	16.00	19.68	15.62
4 8.			5 4	12.00		11.78	5 4	16.08	19.78	15.70
3 8.		8.31	3	12.15	15.02	11.92	3	16.23	19.96	15.84
2 8.	57 10.50		1	12.23		12.00	1	16.31	20.00	15.90
	64 10.7		0	12.38		12.14	0	16.46		16.06
0.9859 8.	71 10.8	8.58	0.9809	12.46	15.40	12.22	0.9759	16.54	20-33	16.13
8 8.	79 10.91	8.66	8	12.54		12.30	8	16.62	20.43	16.21
6 8.	100	8.80	6	12.69		12.37	6	16.77	20.61	16.35
5 9.0			5 4	12.77	15.77	12.51	5 4	16.85	20.71	16.43
3 9-	14 11.35	9.00	3	12.92	15.96	12.66	3	17.00	20.89	16.57
1 9.			2 1	13.00	16.05	12.74	2	17.08	20.99	16.65
0 9.	2 2		0	13.15		12.89	o	17.25	21.19	16.81
0.9849 9.			0.9799	13.23		12.96	0.9749	17-33	21.29	16.89
7 9-		9-35	8 7	13.31	16.43	13.03	8 7	17.42	21.49	16.97
6 9.	64 11.96	9.49	7 6	13.46	16.61	13.18	6	17.58	21.59	17:13
5 9-			5 4	13.54	16.70	13.26	5 4	17.75	21.69	17.20
3 9.1	86 12.22	9.71	3	13.69	16.89	13.40	3	17.83	21.89	17 37 17.46
2 9-9 1 10-0			2	13.77	16.98	13.48	2	17.92	21.99	17-54
0 10.0			0	13.92	17-17	13.63	0	18.08	22.18	17.61
0.9839 10.1	15 12.58		0.9789	14.00	17.26	13.71	0.9739	18.15	24.27	17.68
8 10.: 7 10.:	23   12.68		8	14.09	17-37	13.79	8 7	18.23	22.46	17.76
6 10.	38 12.87	10.20	7 6	14-27	17-59	13.96	6	18.38	22.55	17.90
4 10.5			5 4	14.36	17.70	14.04	5 4	18.46	22.73	17.97
3 10.0	62   13.15	10.44	3	14-55	17.92	14.23	3	18.62	22.82	18.13
1 10.			2	14.64	18.03	14.32	2	18.69	22.92	18.27
0 10.			0	14.82	18.25	14.48	0	18.85	21.10	18.34

HEHNER'S TABLE FOR CALCULATING ALCOHOL FROM SPECIFIC GRAVITY—(Continued)

	Abs	olute Ale	cohol.		Abs	olute Ale	cohol.		Abso	lute Alc	ohol.
Spec. Grav. at r5.6° C.	Per Cent by Weight	Per Cent by Vol- ume.	Grams per 100 cc.	Spec. Grav. at 15.6° C.	Per Cent by Weight	Per Cent by Vol- ume.	Grams per 100 cc.	Spec. Grav. at 15.6° C.	Per Cent by Weight	Per Cent by Vol- ume.	Gram per 100 ce
0-9729	18.92	23.19	18.41	0.9679	22.92	27-95	22.18	0.9629		0	25.6
8	19.00	0 -	18.48	8	23.00	28.04	22.26	8	26.67	32.34	25.6
7 6	19.08		18.56	6	23.08	28.13	22.33	7 6	26.73	32.42	25-7
5	19.25	23.58	18.73	5	23.23	28.31	22.47	5	26.87	32.58	
4	19.33	23.68	18.80	4	23:31	28.41	22.54	4	26.93	32.65	25-9
3	19-42	23.78	18.88	3	23-38	28.50	22.61	3	27.00	32-73	25-9
ī	19.50		18-95	1	23.46	28.59	22.69	1	27.07	32.81	26.0
0	19.67			0	23.62	28.77	22.83	0	27.21	32.98	26.1
0.9719	19-75			0.9669	23.69	28.86		0.9619	27.29		
7	19.83		19.27	8 7	23-77	28.95	22-97	8 7	27.30		26.3
6			19.44	6	23.92	29.13	23.11	6		33-23	26.4
5	20.08	24.58	19.51	5	24.00	29.22	23.19	5	27.57	33-39	26.5
4	20.17	24.68	19-59	4	24.08		23-27	4	27.64	33-48	26.5
3 2	20.25	24.78	19.66	3 2	24.15	29-40	23-33	3 2	27.71	33-56	26.6
1	20.42		19.83	1	24-31	29.58	23.48	ī	27.79	33-64	26.7
0	20.50	25-07		0	24.38		23-55	0	27-93	33.81	26.8
0.9709	20.58	25.17	19.98	0.9659	24.46		23.62	0.9609	28.00	33.89	26.9
8	20.67	25-27	20.07	8	24.54	29.86	23-70	8	28.06		26.9
6	20.83	25-47	20.22	6	24.69	30:04	23.77	6	28.19	34.04	27-0
5	20.92	25-57	20.30	5	24-77	30.13	23.91	5	28.25	34.18	27.1
4	21.00		20.33	4	24.85	30.22	23.99	4	28.31	34-25	27-1
3 2	21.15	25.76	20.40	3 2	24.92	30.40	24.05	3 2	28-37	34-33	27.2
1	21.23	25-95	20.59	1	25.07	30.48	24.19	1	28.50	34-47	27-3
0	21.31	26.04	20.67	0	25.14	30-57	24.26	0	28.56	34-54	27-4
.9699	21.38		20.73	0.9649	25.21	30.65	24-32	0.9599	28.62	34.61	27-4
7	21.54	26.31	20.89	7	25.36	30-73	24-39	7	28.75	34.76	27-5
6	21.62	26.40	20.96	6	25-43	30.90	24-53	6	28.81	34.83	27.6
5	21.69		21.03	5	25.50	30.98	24.59	5	28.87	34.90	27.7
4	21.77	26.58	21.11	3	25.57 25.64	31.07	24.66	3	28.94	34-97	27.7
2	21.92	26.77	21.25	2	25.71	31.23	24.79	2	20.07	35.05	27.8
1	22.00	26.86	21.33	1	25.79	31.32	24.86	1	29-13	35.20	27.9
0	22.08	26.95	21.40	0	25.86	31.40	24-93	0	29.20	35.28	28.0
.9689	22.15	27.04	21.47	0.9639	25.93	31.48	24.99	0.9589	29.27	35-35	28.0
7	22.23	27.22	21.54	7	26.07	31.57	25.00	7	29.33	35-43	28.1
6	22.38	27-31	21.68	6	26.13	31.72	25.18	6	29-47	35.58	28.2
5	22.46	27.40	21.76	5	26.20	31.80	25.23	. 5	29-53	35.00	28.3
4	22.54	27.49	21.83	4	26.27	31.88	25-30	4	29.60	35-74	28-3
3	22.62	27.59	21.90	3 2	26.33	31.90	25.36	3	29-73	35.81	28.4
î	22.77	27-77	22.01	1	26.47	32.11	25.49	1	29.80	35-97	28.5
0	22.85	27.86	22.12	0	26.53	32.19	25-55	0	29.87	36.04	28.6

HEHNER'S TABLE FOR CALCULATING ALCOHOL FROM SPECIFIC GRAVITY—(Continued)

	Abs	olute Ale	ohol.	Core	Abs	olute Ale	ohol.	Care	Abso	olute Alc	ohoL
Spec. Grav. at 15.00 C.	Per Cent by Weight	Per Cent by Vol- ume.	Grams per 100 cc.	Spec. Grav. at 15.6° C.	Per Cent by Weight	Per Cent by Vol- ume.	Grams per 100 cc.	Spec. Grav. at 15.6° C.	Per Cent by. Weight	Per Cent by Vol- ume,	Grams per 100 cc
0.9579	29.93	36.12	28.67	0.9529	32.94	39-54	31.38	0.9479	35-55	42-45	33-70
8	30.00	36.20	28.73	8	33.00		31.43	8	35.60	42.51	33-75
7 6	30.06		28.78	7 6	33.06		31.48	7 6	35.65		33-79
5	30.11	36.32	28.88	5	33.12	39.74	31.53	5	35-70	42.67	33.88
4	30.22		28.92	4	33.24	39.87	31.63	4	35-75 35.80		33.92
3	30.28		28.98	3	33.29		31.69	3	35.85	42.78	33.97
2		36.57	29.03	2	33-35	40.01	31.74	2	35.90	42.84	34.01
1	0 4		29.08	1	33-41	40.07	31.80	1	35-95	42.89	34.05
0	30-44	36.70	29.13	0	33-47	40.14	31.86	0	36.00	42-95	34.09
0.9569	30.50	36.76	29.18	0.9519	33-53	40.20	31.91	0.9469	36.06	43.01	34.14
8	30.50	36.83	29.23	8	33-59		31.96	8	36.11	43.07	34.09
7	30.61			7 6	33.65		32.01	7	36.17		34-24
6	0		29-33	11	33-71	40.40	32-07	6	10		34-28
5			29.38	5 4	33.76	40.47	32.12	5 4	36.28		34-34
- 3		37.14	29.48	3	33.88	40.60	32.22	3	36.39		34.44
2				2	33-94	40.67	32.27	2	36.44		34.48
1	30.94	37-27	29-58	1	34.00		32-32	1	36.50		34-54
0	31.00	37-34	29.63	0	34-05	40.79	32-37	0	36.56	43-56	34-58
0.9559	31.06	37-41	29.69	0.9500	34.10	.40.84	32.41	0.9459	36.61	43.63	34.6
-8	31.12		29.74	8	34-14	40.90	32.45	8	36.67	43.69	34.69
7		37-55	29.81	7	34.19		32.49	7	36.72	43-75	34-73
6	10	37.62	29.86	6	1 24		32-54	6			34-79
5			29.91	5 4	34-29		32.59	5	36.83	43.87	34.8
3			30.03	3	34.38		32.67	3	36.94		34-9
2				2	34-43		32.71	2	37.00	44.06	34-9
1	31.56	37-97	30.14	1	34.48		32-75	1	37.00	44.12	35.0
C	31.62	38.04	30.20	0	34-52	41.32	32.79	0	37-11	44.18	35.0
0.9549		38.11	30.26	0.9499	34-57		32.84	0.9449			35.12
8		38.18		8	34.62	41.42	32.88	8	37-22	44.30	
7	31.81			7 6	34.67		32-92	7 6	37.28		
5	10	0		5	34-71		32.90	5	37-33		35.2
4		0	30.53	4	34.81	41.63	33.04	4	37-44		35-3
3		38.53	30.59	3		41.69	33.09	3	37-50		35-4
2		38.60	30.64	2	34.90	41.74	33.13	2	37.56	44.67	35-4
	10.			1	34.95		33:17	1	37.61	44-73	35-5
0	32.25	38.75	30-77	0	35.00	41.84	33.21	0	37.67	44-79	35-50
0.9539	32.31		30.81	0.9489	35.05		32.26	0.9439	37-73	44.86	35.60
8	32-37		30.87	8	35.10		33.30	8	37-78	44.98	35.65
7 6	32.44			7 6	35-15		33-34	7 6	37-83	44.98	35-79
5	32.50		30.99	5	35-25	42.00	33-39	5	37.89		35.75
4	32.62		31.10	4	35-25	7.	33.48	4	37-49	45.16	35.85
3	32.69		31.15	3	35-35	42.23	33-53	3	38.06		35.90
2	32.75	39-32	31.20	2	35-40	42.29	33-57	2	38.11	45.28	35-95
	Q.	1 20 40	31.26	1		42.34	33.61	1	38.17	40 24	36.00
1 0	32.81	39-40	31.32	o	35-45	42.40	33.65	0	38.22	45-41	36.04

HEHNER'S TABLE FOR CALCULATING ALCOHOL FROM SPECIFIC GRAVITY—(Continued)

	Abs	olute Ald	ohol.		Abs	olute Ale	cohol.		Abso	lute Alc	ohol.
Spec. Grav. at 15.6° C.	Per Cent by Weight	Per Cent by Vol- ume.	Grams per 100 cc.	Spec. Grav. at 15.6° C.	Per Cent by Weight	Per Cent by Vol- ume.	Grams per 100 cc.	Spec. Grav. at 15.6° C.	Per Cent by Weight	Per Cent by Vol- ume.	Grams per 100 cc
0.9429	38.28	45-47	36,08	0.9379	40.85	48.26	38.31	0.9329	43.29		40.3
8	38.33	45-53	36,13	7	40.90	48.32	38.35	8 7	43-33	50.92	40.4
6	38.44	45.65	36.23	6	41.00	48.43	38.44	6	43.43		40.5
5	38.50	45-71	36.28	5	41.05	48.48	38.48	5	43.48		40.5
4	38.56	45-77	36.33	3	41.10	48.54	38.52	3	43-52	51.12	40.5
2	38.67	45.89	36.43	2	41.20		38.62	2	43.62	51.22	40.6
0	38.72 38.78	45-95	36.48 36.53	0	41.25		38.66	0	43.67	51.27	40.7
0.9419			36.57 36.62	0.9369	41.35		38.74 38.78	0.9319	43.76 43.81	51.38	40.7
7	38.94	46.20	36.67	7 6		48.91	38.82	7	43.86		40.8
6	MA		36.72				38.87	6	43-90		40.8
5			36.80	5 4	41.55	49.02	38.91	5 4	43.95	51.58	40.9
3	39.15	46.42	36.85	3		49.13	38.99	3	44.05	51.68	41.0
1	39.20	46.48	36.89	2	41.70	49.18	39.04	2 I	44.09	51.72	41.0
0				0	41.80		39.13	0	44.18		41 1
9409			37.02	0.9359	41.85	49:34	39.17	0.9309	44-23	51.87	41.1
7	39-45	46.75	37-11	7 6		49-45	39.25	7	44-32		41.2
6	03.0		37.15	5		49 50	39.30	6	44.41	52.01	41.2
5			37-23	4	42.10		39.38	5 4	44.46		41.3
3			37-27	3		49.66		3	44-50		41.4
2		- 0	37-32	2	42.19		39.46	2	44-55		41.4
•				0	42.29		39-54	0	44.64		41.5
0.9399				0.9349	42.33	49.86	39.58	0.9299	44.68		41.5
7	39-95	47-29	37-53	7	42.43	49.96	39.66	7	44.77	52.44	41.6
	100000000000000000000000000000000000000		37.58	5		50.01	39.79	5	44.82	52.48	41.6
54				4	42.57	50.11	39-78	4	44.91	52.58	41.7
3			37-71	3		50.16	39.82	3	44.96	52.63	41.7
2				2	42.67	50.21		2	45.00		41.8
c	- M			0			39-94	0	45.09		41.8
0.9389	40.35		37.88	0.9339	42.81	50-37	39.98	0.9289	45.14		41.9
8	40.40		37-92 37-96	8 7	42.86		40.02	8 7	45.18		41.9
2		47.89	38.00	7 6		50.52	40.10	6	45.27	52.96	42.0
5			38.05	5	43.00	50.57	40.14	5	45:32		42.0
4			38.13	3	43.10		40.22	3	45-30	53.10	42.1
2	40.70	48.10	38.18	2	43.13	50.72	40.26	2	45-46	53:15	42.1
1			38.22	1 0	43.19	50.77	40.30	0	45-50	53-20	42.2
	40.00	40.41	30/		10.24	3-1-1	134		43-33	334	

# LISTS OF APPARATUS, REAGENTS, AND PRACTICE MATERIAL

### APPARATUS FOR USE IN COMMON

THE following instruments, pieces of multiple apparatus, etc. will be sufficient for a class of thirty members.

- 6 Balances, analytical, each with weights, balanced watch glasses, and camel's-hair brush.
  - 1 Saccharimeter and lamp (Fig. 84).
- 6 Saccharimeter Tubes, glass, 200 mm., enlarged at one end (Fig. 86).
  - 1 Refractometer, Abbé, with thermometer (Fig. 90).
  - 1 Westphal Balance complete with extra float (Fig. 88).
  - I Tintometer, Lovibond (Fig. 106), with slides as follows: Red 5.0, 2.0, 2.0, 1.0, 0.5, 0.2, 0.2, 0.1.

Yellow 10.0, 5.0, 2.0, 2.0, 1.0, 0.5, 0.2, 0.2, 0.1. Blue (see p. 190).

- 6 Microscopes (Fig. 38), each with double nosepiece, 16 and 4 mm. objectives, 10 × eyepiece, 6 × eyepiece, micrometer ruled to 0.01 mm., and stage micrometer ruled to 0.01 mm.
  - 1 Microscope with polarizing apparatus.
- 1 Colorimeter, Schreiner, with extra immersion and graduated tubes (Fig. 107).
  - 1 Lactometer, Quevenne (Fig. 2), and cylinder.
  - 1 Melting-point Apparatus (p. 188).
  - 2 Pipettes, Babcock, 17.6 cc. (Fig. 7).
  - 2 Acid Measures, Babcock (Fig. 11).
  - 18 Dropping Bottles, microscopic (Fig. 40).
  - 1 Generator, Kipp, large.
  - 1 Centrifuge, Babcock (Fig. 12).
- I Kjeldahl Digestion Stand with 13 burners complete (Fig. 33).

- I Kjeldahl Distillation Stand with 13 burners complete (Fig. 34).
- 1 Drying Apparatus for 12 determinations complete as shown in Fig. 26, also 18 glass drying tubes with corks for both ends and 18 exit tubes, 0.5 mm. bore, in corks to fit large end of drying tubes.
  - 2 Water Ovens (Fig. 6).
  - 1 Cork Tongs.
  - 1 Cork Borer Set (Fig. 16).
  - 1 Cork Borer Sharpener (Fig. 17).
  - 2 Mortars, iron (Fig. 25).
  - 1 Food Chopper, Universal (Fig. 24).
  - 1 Funnel, copper, short stem (Fig. 27).
  - 1 Sugar Weighing Dish, nickel (Fig. 85).
- 1 Can, kerosene, galvanized iron, 5 gal., with cock, for water tank (p. 149)
  - 1 pair Forceps (Fig. 93).
- I Sieve with round holes about  $\frac{1}{25}$  in. diam., cover, and receiver (Fig. 23).

Note.—At the date of writing the war conditions are such that the importation of apparatus is difficult or impossible. The refractometer, tintometer, and Westphal balance may be dispensed with without serious detriment to the course. Even the saccharimeter is not absolutely indispensable if a visit can be arranged to a laboratory carrying on sugar polarizations. Substitutes for the multiple Kjeldahl apparatus are described on page 65.

#### APPARATUS FOR INDIVIDUAL USE

In this list are included such pieces of apparatus as are required by each student in carrying out the work of this course, with no allowance for breakage.

- 1 Thermometer, chemical, graduated o-100° C.
- 2 Burettes, 50 cc., one with ball cock, the other with glass stopcock (Fig. 35).
  - 1 Pipette, 50 cc.
  - 1 Pipette, 25 cc.
  - 1 Pipette, 20 cc.

- 1 Pipette, 10 cc.
- 1 Pipette, 5 cc.
- 1 Pipette, 2 cc.
- 1 Pycnometer, 100 cc., with delivery tube to attach to 15 in. condenser (Figs. 99 and 100).
  - 1 Flask, graduated, 500 cc.
  - 1 Flask, graduated, 100 and 110 cc. marks.
  - 1 Flask, graduated, 50 and 55 cc. marks.
  - 1 Bottle, Babcock, milk.
  - 1 Bottle, Babcock, cream, Winton, 10 to 30 per cent.
  - 1 Bottle, Babcock, skim milk, Wagner.
- 1 Flask, flat bottom, ring neck, 1000 cc., with wash bottle fittings complete.
- 1 Flask, flat bottom, ring neck, 500 cc., with double-bore rubber stopper (Fig. 21).
- 1 Flask, flat bottom, ring neck, 300 cc., with single-bore rubber stopper (Figs. 95 and 100).
  - 2 Flasks, Kjeldahl, flat bottom, short neck, 500 cc.
- 2 Flasks, flat bottom, vial mouth, 30 cc., inside diameter large enough to admit delivery tube of Johnson extractor with cork, and 4 corks (Fig. 15).
  - 4 Flasks, Erlenmeyer, 500 cc.
- 2 Flasks, Erlenmeyer, 200 cc., and 2 single-bore rubber stoppers (Fig. 94).
- 1 Flask, filtering, 500 cc., heavy glass with side neck and single-bore rubber stopper (Fig. 19).
  - 2 Beakers, 400 cc.
  - 2 Beakers, 250 cc.
  - 2 Watch-glasses, 90 mm.
  - 2 Funnels, 6.5 cm. diam., for 11 cm. filters.
  - 2 Funnels, 11 cm. diam., for 18.5 cm. folded filters.
- 2 Funnels, separatory, Squibb's, pear shape, glass stoppered, 125 cc.
- 2 Condensers, Liebig's, all glass, 15 in., and 1 single-bore rubber stopper to fit top (Figs. 21, 25, etc.)
  - 2 Fat Extractors, Johnson, outer tubes 175 mm. long (not

including delivery tube), 26 mm. inside diam. (Fig. 15), also 4 corks to fit top.

- <sup>2</sup> Fat Extractors, Johnson, inner tubes, 135 mm. long, 22 mm. outside diam., not flanged at top (Fig. 28).
  - 1 Tube, filtering, for Gooch crucibles, 28 mm. diam. (Fig. 19).
  - 1 Tube, melting-point, capillary, closed at one end (p. 188).
  - 6 Slides, microscopic 3 × 1 in.
  - 12 Cover Glass Circles, microscopic, No. 2, 3 in. diam.
- 4 Dishes, crystallizing, glass, low form, 38 mm. high, 60 mm. diam.
- <sup>2</sup> Cylinders, fat, glass, flat bottom, 15 mm. high, 10 mm. diam. (Fig. 93).
  - 2 Test-tubes.
- 2 Bottles, weighing, flat bottom, without neck, ground stoppered, 75 mm. high, 40 mm. diam.
- 4 Bottles, narrow mouth, glass stoppered, 250 cc. (Fig. 93). Mouth must be large enough to admit any of the fat cylinders listed above.
  - 1 Bottle, wide mouth, 8 oz., glass stoppered for subsample.
  - 1 Bottle, wide mouth, 8 oz. (Fig. 21).
  - 1 Bottle, wide mouth, 4 oz. (Fig. 21).
  - 1 Tube, bulb, connecting (Fig. 21).
  - 3 Tubes, connecting as shown in Fig. 21.
- 1 Desiccator, Scheibler, 150 mm. diam., with wire gauze disk (Fig. 4).
  - 2 Crucibles, Gooch, porcelain, 35 mm. diam. (Fig. 19).
  - 2 Crucibles, porcelain, 40 mm. diam. (pp. 63 and 73).
- 12 Dishes, tinned lead (bottle caps),  $2\frac{1}{2}$  in. diam.,  $1\frac{1}{16}$  in. high (Fig. 3).
  - 2 Burners, Bunsen.
- 2 Supports, iron, rectangular base, rod 36 in. high (Figs. 15, etc.).
  - 2 Rings, iron, for supports, 4 in. diam. (Figs. 15, etc.).
- 2 Clamps, iron, double jaws, separate fasteners for supports (Figs. 15, etc.).
  - 1 Support, wood, for 2 burettes (Fig. 35).

- 1 Support, wood, for 2 separatory funnels (Fig. 104).
- 1 Pump, filter, Chapman, 3\frac{3}{4} in., with coupling (Fig. 19).
- 1 Pan, enameled, quart, bottom at least 5 in. (pp. 132 and 186).
- 1 Cup, enameled, pint (water bath), and copper ring with 2 in. hole (p. 16).
  - 2 Wire Gauze Squares, 5 in.
  - 2 Sheet Iron Squares, 5 in. (Fig. 15).
  - 1 sq.ft. Asbestos paper.
  - 1 Spoon, aluminum or tin, small.
  - 6 ft. Tubing, rubber, for lamps and condensers.
  - 2 ft. Tubing, rubber, thick, for suction apparatus (Fig. 19).
- 2 in. Tubing, rubber, thick, 1 in. inside diam., for Gooch crucibles (Fig. 19).

#### REAGENTS

The quantities given are those needed by a class of thirty students or else the minimum advisable to purchase.

- 10 lbs. Acid, acetic, glacial, c.p.
- 24 lbs. Acid, hydrochloric, sp.gr. 1.20, c.p.
- 1 lb. Acid, molybdic, c.p.
- 7 lbs. Acid, nitric, c.p.
- 100 grams Acid, phospho-molybdic crys., c.p.
- 1 lb. Acid, phosphoric, 85%, c.p.
- 27 lbs. Acid, sulphuric, c.p.
- 9 lbs. Acid, sulphuric, c.p., free from nitrogen.
- 9 lbs. Acid, sulphuric, common, sp.gr. 1.82-1.83.
- 1 oz. Acid, tannic.
- 3 lbs. Alcohol, amyl, c.p.
- 2 gals. Alcohol, ethyl, 95%.
- 1 lb. Aluminum sodium sulphate, tech.
- 1 lb. Ammonium chloride, c.p.
- 8 lbs. Ammonium hydroxide, c.p.
- 2 lbs. Asbestos, amphibole, for Gooch crucibles, washed with acid and guaranteed to retain copper suboxide and barium sulphate.
  - I lb. Asbestos wool for milk analysis.

- 1 lb. Barium chloride, c.p.
- 1 lb. Bromine.
- 10 lbs. Calcium carbonate, marble, lumps.
- 1 lb. Carbon disulphide.
- 2 lbs. Chloroform.
- 10 grams Citral, c.p.
- 1 yd. Cloth, cheese.
- 1 yd. Cloth, white woolen, nun's veiling.
- 1/4 lb. Cochineal, powdered.
- 1 lb. Copper sulphate, crys., c.p.
- Ilb. Cotton wool.
- 2 oz. Diamond ink.
- 3 lbs. Ether, absolute.
- 20 lbs. Ether, conc., U.S.P.
- 1 lb. Formaldehyde, 40%.
- 5 lbs. Fuller's earth.
- ı gal. Gasolene.
- I lb. Glycerine.
- ½ lb. Iodine, resublimed.
- I lb. Lead acetate, c.p.
- 5 lbs. Lead subacetate, c.p., dry.
- 5 sheets Litmus paper, neutral.
- 1 oz. Mercury, oxide, red, c.p.
- 1 oz. Phenolphthalein.
- 10 grams Phenylenediamine, meta, hydrochloride.
- ½ lb. Potassium bisulphate, crys., c.p.
- 1 lb. Potassium iodide.
- ½ lb. Potassium oxalate.
- ½ lb. Potassium permanganate, crys., c.p.
- I lb. Pumice stone, granulated (about I mm.).
- I oz. Silver nitrate.
- 5 lbs. Sodium carbonate, crys., c.p.
- 2 lbs. Sodium hydroxide, electrolytic.
- 1 lb. Sodium hydroxide, from Na.
- 5 lbs. Sodium hydroxide, granular, 95%.
- 5 lbs. Sodium potassium tartrate, crys., c.p.

I lb. Sodium sulphide.

I lb. Sodium thiosulphate, crys., c.p.

1 lb. Sodium tungstate.

1 lb. Sulphur, flowers.

I spool Thread, linen, white, No. 25.

5 sheets Turmeric paper.

1 oz. Uranium acetate.

I oz. Vanillin.

I lb. Zinc, granulated, 20 mesh.

to lbs. Zinc, mossy, coml.

#### MATERIAL FOR LABORATORY PRACTICE

Most of the materials may be obtained of the grocer, druggist, milkman, or feed dealer. The coal-tar colors, vanillin, coumarin, lemon oil, terpeneless lemon oil, potato starch, and cassava starch would better be ordered with the reagents from a dealer in laboratory supplies. The quantities are sufficient for thirty students.

Dairy Products. 6 lots of 1 qt. Milk, 1 pt. Skim Milk, and 1 pt. Cream. 2 lbs. Butter.

Meat. 6 lots of 2½ lbs. Hamburg Steak.

Mill Products, etc. 1 lb. each of Wheat Flour, Graham Flour, Rye Flour, Buckwheat Flour, Corn Meal (whole kernel), Oat Meal, Rice, Hominy, Cream of Wheat, Force, Corn Flakes, Grape Nuts, Beans, Wheat Bran, Rye Bran, Linseed Meal, and Cotton Seed Meal.

Baking Powder. 1 lb. each of Royal, Horsford's, and K. C., Calumet, or O. K.

Starch. 1 lb. each of Corn, Potato, and Cassava Starch.

Unground Grains and Seeds. 1 lb. each of Wheat, Rye, Corn (maize), Oats, Buckwheat, Peas, Cotton Seed, and Flax Seed.

Spices. ½ lb. each of ground and unground Black Pepper, Cayenne Pepper, Cinnamon (cassia), and Ginger.

Alkaloidal Products. ½ lb. each of Tea, Cocoa Beans, and Cocoa. 2 lbs. of Coffee Beans, 2 lbs. Ground Coffee. 1 lb.

each of Roasted Peas and Wheat ground for mixing with ground coffee.

Saccharine Products. 10 lbs. Sugar, granulated. 2 qts. each of Molasses and Karo Syrup. 4 lbs. Honey, strained. 1 qt. Raspberry or Strawberry Syrup. 10 grams each of Amaranth, Ponceau 3R, Erythrosin, Orange I, Naphthol Yellow S., 1 oz. Cudbear or other lichen color.

Fats and Oils. 2 lbs. of Oleomargarine. 1 lb. each of Cocoanut Oil, Beef Tallow, Lard, and Cocoa Butter. 1 pt. each of Olive Oil, Cotton Seed Oil, Peanut Oil, Sesame Oil, and Rape Oil.

Fruit Products. 6 pint bottles each of Sweet Cider, sterilized, and Fermented Cider. 1 qt. of Cider Vinegar.

Extracts. 2 qts. Vanilla Extract, pure (or ½ lb. Vanilla Beans for making same). 2 qts. Imitation Vanilla Extract made from vanillin and coumarin, colored with caramel (or 1 oz. each of the ingredients for making same). 1 qt. Lemon Extract (or 2 oz. Lemon Oil for making same), 1 pt. Terpeneless Lemon Extract (or 10 grams Terpeneless Lemon Oil for making same).

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